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Synthesis and Study of New Deep Eutectic Solvents on the Principles of Green Chemistry Based on Pentaerythritol, Choline Chloride and Urea

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Abstract. The paper presents the results of synthesis and investigation of binary and ternary systems of deep eutectic solvents based on pentaerythritol, urea and choline chloride. Investigations of the DES systems by IR spectroscopy allowed to assume the mechanism of donor-acceptor interaction of their components with the formation of a molecular complex. The presence of a large number of hydrogen bonds leads to a decrease in the melting/crystallisation temperature of eutectic DES compositions, which is explained by intermolecular forces and self-association at donor-acceptor interaction of components. This interaction leads to a decrease in the lattice energy between the DES molecules and the destruction of the solid crystal structures of the DES components. As a result, less energy is required to melt the molecules than to melt the individual components.

Keywords: deep eutectic solvents, donor-acceptor interaction, eutectic, phase diagram, two-component and three-component system.

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Синтез и исследование новых глубоких эвтектических растворителей на принципах зеленой химии на основе пентаэритрита, хлорида холина и карбамида

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Аннотация. В работе представлены результаты синтеза и исследования бинарных и тройной систем глубоких эвтектических растворителей на основе пентаэритрита, карбамида и хлорида холина. Исследования систем ГЭР методом ИК-спектроскопии позволили предположить механизм донорно-акцепторного взаимодействия их компонентов с образованием молекулярного комплекса. Присутствие большого количества водородных связей приводят к снижению температуры плавления/кристаллизации эвтектических составов ГЭР, что объясняется межмолекулярными силами и самоассоциацией при донорно-акцепторном взаимодействии компонентов. Такое взаимодействие приводит к снижению энергии решетки между молекулами ГЭР и разрушению твердых кристаллических структур компонентов ГЭР. В результате этого для плавления молекул требуется меньшая энергия, чем для плавления отдельных компонентов.

Ключевые слова: глубокие эвтектические растворители, донорно-акцепторное взаимодействие, эвтектика, фазовая диаграмма, двухкомпонентная и трехкомпонентная система.

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Цитирование: Шолидодов М. Р., Алтунина Л. К., Сайденцаль А. Р., Козлов В. В., Кувшинов В. А. Синтез и исследование новых глубоких эвтектических растворителей на принципах зеленой химии на основе пентаэритрита, хлорида холина и карбамида. Журн. Сиб. федер. ун-та. Химия, 2025, 18(1). С. 135–146. EDN: SWBHED

Introduction

Currently, green chemistry has become extremely important due to environmental pollution and health and safety hazards for employees working with chemicals in industry and laboratories. In this context, deep eutectic solvents (DES) have attracted much attention from scientific and industrial communities due to their unique physicochemical properties such as ease of preparation, biodegradability, thermal stability, non-toxicity, etc. They are considered as promising alternatives to traditional organic solvents and ionic liquids. Over the last two decades, deep eutectic solvents have evolved from an interesting laboratory phenomenon to promising solvents widely used in many fields of science.

For the first time, DESs were obtained by Abbott [1–7]. DESs are usually liquids consisting of two, three or more compounds. These compounds can self-combine through hydrogen bonding to

form a eutectic mixture with a melting point lower than that of each individual component [8–10]. The decrease in melting temperature is due to the formation of intermolecular hydrogen bonds between DES molecules to form complex compounds [11–14].

Most of the components for the synthesis of DESs are cheap and readily available. For example, the most commonly used choline chloride is a non-toxic, biodegradable and economical quaternary ammonium salt that can rapidly form DESs with various hydrogen bond donors [15].

The unique chemical properties and application advantages of DES, such as tunable solubility and high chemical stability, make it widely used in various scientific and technological fields such as chemical synthesis, energy storage, pharmaceutical preparation, material modification, substance extraction and so on. Recently, attempts to use DES as a green alternative to surfactant solutions in conventional chemical enhanced oil recovery methods have been reported in the literature.

This study presents the results of synthesis and investigation of new binary DES systems based on pentaerythritol, urea and choline chloride, as well as a ternary system based on these components.

Experimental

For synthesis of new three two-component DESs ("pentaerythritol – choline chloride"; "pentaerythritol – urea" and "choline chloride – urea") and one three-component DES system "pentaerythritol – urea – choline chloride", chemically pure substances were used: pentaerythritol, urea, and choline chloride (Table 1).

Table 1. Physicochemical characteristics of the initial components of DES

Reagents	Chemical formula	Molecular weight, g/mol	Melting point, °C
Pentaerythritol	$C_5H_{12}O_4$	136.0	253.0
Urea	$(NH_2)_2CO$	60.0	132.5
Choline chloride	$C_5H_{14}ONCl$	139.5	302.0

In this work, DES systems were investigated: DES based on the binary system of pentaerythritol and choline chloride; DES based on the binary system of pentaerythritol and urea; DES based on the binary system of choline chloride and urea; DES based on the ternary system of pentaerythritol, urea and choline chloride.

Synthesis of binary DES systems ("pentaerythritol – choline chloride", "pentaerythritol – urea", "choline chloride – urea") was carried out as follows: DES components were initially dried in a desiccator to constant weight. Then in a mortar grinded pre-weighed on analytical scales suspensions of components of two-component DES systems in different molar ratios (from 1:10 to 10:1 mole fractions) to complete homogeneity. The obtained mixtures were poured into clean dry flasks and heated on a sand bath with constant stirring until the formation of transparent melts. The flasks with melts were placed in a thermostat heated to 80 °C for 4–6 hours and then cooled to room temperature.

To determine the crystallization temperature in the range from room temperature to 30 °C below zero, the samples were placed in a thermostat (cryostat) filled with a cooling mixture (ethanol) with a temperature 3–5 °C below the expected crystallization temperature.

To determine the presence of hydrogen bonds in the binary and ternary DES systems, infrared spectroscopy was used on a Nikolet 5700 FTIR spectrometer with Raman module (Thermo Electron Corporation, USA). This method is based on the measurement and analysis of the light absorption spectrum in the IR region, which is caused by vibrations of atomic bonds in the molecule.

The solubility of the ternary system "pentaerythritol – urea – choline chloride" was measured by dissolving DES in water until it precipitates, with further incubation of the aqueous solution at a certain temperature, separation of the precipitate and determination of the amount of dissolved substance.

Density of solutions of compositions and formation fluids was determined by pycnometric method and with the help of density meter EASY D 40.

Determination of pH values was carried out using a microprocessor laboratory pH-meter manufactured by HANNA Instruments.

Results and Discussion

According to the method described above in the section "Experimental", binary mixtures of DESs in different molar ratios of components were synthesised and obtained: "pentaerythritol – choline chloride", "pentaerythritol – urea", "choline chloride – urea". The measured melting/crystallization temperatures allowed us to construct phase equilibrium diagrams of binary systems: the dependence of melting (crystallization) temperature on the molar ratio of components.

Fig. 1 show the phase equilibrium diagrams of DES: a – DES based on pentaerythritol and choline chloride; b – DES based on pentaerythritol and urea, as well as c – DES based on choline chloride and urea.

The diagrams show that the binary systems of DES based on "pentaerythritol – choline chloride" (Fig. 1a), "pentaerythritol – urea" (Fig. 1b) and "choline chloride – urea" (Fig. 1c) are characterised by one eutectic point (the lowest point of the solidus line marked with the symbol 'E') (Fig. 1a, b, c).

The melting temperature of the mixture at eutectic points is much lower than the melting temperature of the individual initial components of the mixture (pentaerythritol – 253 °C, choline chloride – 302 °C, urea – 132 °C). Melting/crystallisation temperature for the binary system of DES based on pentaerythritol and choline chloride in the molar ratio of components 50,0 % mol.

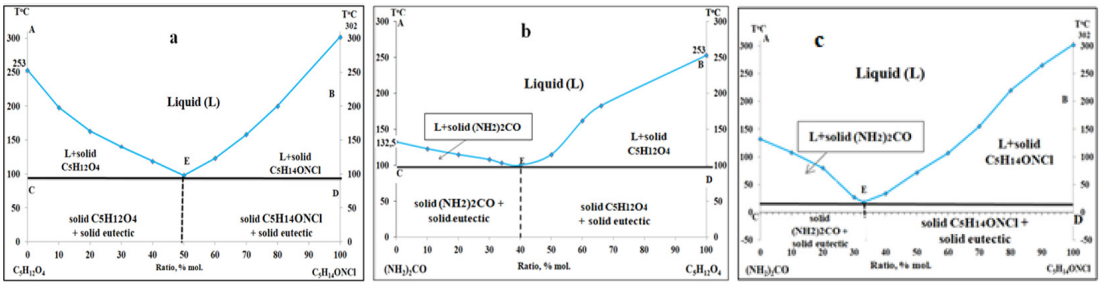

Fig. 1. Phase equilibrium diagram of the DES system: a – DES based on pentaerythritol and choline chloride; b – DES based on pentaerythritol and urea; c – DES system based on choline chloride and urea

pentaerythritol and 50,0 % mol. choline chloride is 98 °C, for the binary system of DES based on pentaerythritol and urea in the molar ratio of components 40,0 % mol. pentaerythritol and 60,0 % mol. urea is 96 °C, and for the binary system of DES on the basis of choline chloride and urea in the molar ratio of components 33,0 % mol. choline chloride and 67,0 % mol. urea is 18 °C. The density of the mixtures of binary DES systems is in the range of 1,2158 to 1,2458 g/cm³.

The study of phase equilibrium diagrams of binary DES systems: DES based on pentaerythritol and choline chloride; DES based on pentaerythritol and urea, as well as DES based on choline chloride and urea allowed theoretically calculate the eutectic composition and synthesise the ternary DES system "pentaerythritol – urea – choline chloride" (Fig. 2, Table 2).

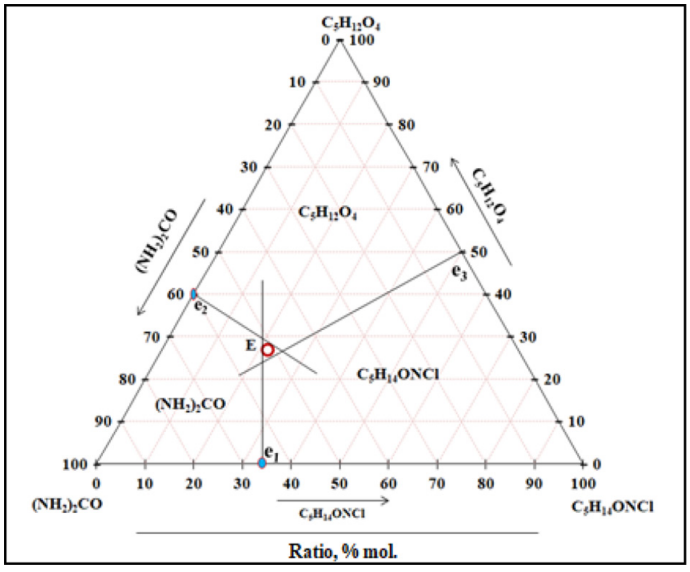

Fig. 2. Phase equilibrium diagram of the DES system based on pentaerythritol, urea and choline chloride

Table 2. Physicochemical characteristics of binary and ternary DES systems based on pentaerythritol, urea and choline chloride

DES based on	Hydrogen bond donor	Hydrogen bond acceptor	Ratio of components, % mol.	Solidification/ crystallisation temperature, °C	Density, g/cm^3
Pentaerythritol and choline chloride	Pentaerythritol	Choline chloride	50.0:50.0	98	1.2458
Pentaerythritol and urea	Pentaerythritol	Urea	40.0:60.0	96	1.2346
Choline chloride and urea	Urea	Choline chloride	67.0:33.0	18	1.2158
Pentaerythritol, urea and choline chloride	Pentaerythritol and urea	Choline chloride	27.0:51.5:21.5	14 below zero	1.2387

The ratio of components in such theoretically calculated three-component DES system based on pentaerythritol, urea and choline chloride was 27,0 % mol. of pentaerythritol, 21,5 % mol. of choline

chloride and 51,5 % mol. of urea. Determination of the melting point of the theoretically calculated and synthesised eutectic mixture showed that it is characterised by a lower temperature than the melting point of each of the binary systems of eutectic composition and is 14,5 °C below zero.

The presence of one eutectic point and the absence of other extrema on the solidus line in binary and ternary systems indicates the absence of formation of chemical compounds: the system exists as a mixture of components with donor-acceptor interaction.

Fig. 3–6 show the results of IR spectroscopy study of binary and ternary DES systems and initial DES components.

The IR spectra of DES samples based on the binary systems "pentaerythritol – choline chloride", "pentaerythritol – urea" and "choline chloride – urea" and the ternary system 'pentaerythritol – urea – choline chloride' show that the strongest and widest absorption band is in the region of H–-O and N–-H bond stretching between 3100–3600 cm^{-1}, as shown in Fig. 3–6. This is due to the fact that the primary amide groups in compounds in the initial state (in this case urea) give two narrow bands in this region. When a hydrogen bond is formed, the vibrational frequency decreases and the absorption bands broaden to a single broad band. Other species of amide group (in this paper in choline chloride) in the initial state give only one narrow band in these absorption regions. When a hydrogen bond is formed, the vibrational frequency decreases and the bands broaden [16].

According to the authors [17–18], the absorption bands in the interval 3650–3200 cm^{-1} characterize the presence of -OH group, and in DES these absorption bands shift to 3500–2500 cm^{-1}. This is due to the fact that during the formation of hydrogen bonding, the bonding strength in the O-H group decreases due to the recoil of electrons, and the absorption bands may shift [19–20]. The stronger the resulting interaction, the lower the frequency of vibrations. The absorption bands of hydroxyl groups (in this work pentaerythritol) formed by hydrogen bonds have an increased width and are shifted to a lower frequency region (3550–3450 cm^{-1}) compared to the original substance (Fig. 3). The shift and broadening of the absorption band in the IR spectra of DESs, which include

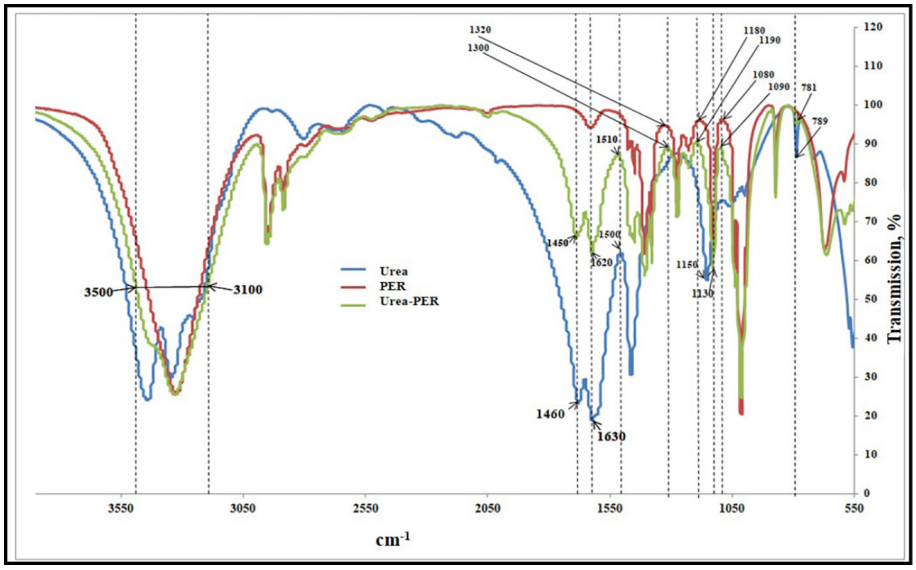

Fig. 3. IR spectra of Urea, PER and DES based on Urea-PER

the O-H group, indicates that a strong hydrogen bond was formed between the molecules directly with the O-H group.

Peak shifts at 3442, 1630, 1460, 1090, 1190 and 789 cm^{-1}, which are attributed to O-H, N-H and C+CH_2-O bond stretching (3442, 1690 and 1630 cm^{-1}, respectively), are observed for the binary DES system "urea – pentaerythritol". The peaks at 1460, 1630, 1500 and 783 cm^{-1} are related to N-H bond breaking, CH_2 bending, C+CH_2-O stretching and C–C valence vibration, respectively.

Fig. 3 shows the IR spectrum of the binary system of DES based on urea and pentaerythritol (PER).

Importantly, in urea- and pentaerythritol-based DESs, the shift of the C+CH_2-O peak and stretching vibrations from 1092 to 1084 cm^{-1}, 1190 to 1180 cm^{-1}, 1320 to 1300 cm^{-1}, and the shift of the N-H peak stretching vibrations from 1450 to 1460 cm^{-1}, 1630 to 1620 cm^{-1}, 1500 to 1510 cm^{-1}, indicates that more stable hydrogen bonds are formed between urea and pentaerythritol [21–22].

Fig. 4 shows the mechanism of donor-acceptor interaction of DESs based on urea and pentaerythritol. As a result of donor-acceptor interaction of components of DES on the basis of urea and pentaerythritol, a molecular complex compound is formed, in which urea is an acceptor of electron pairs of the donor, and pentaerythritol is a donor of electron pairs (Fig. 4).

Fig. 5 shows the IR spectrum of the binary (6 a) DES system based on pentaerythritol and choline chloride (ChCl) and (6 b) of the binary DES system based on urea and choline chloride. From the IR spectrum, it can be seen that the binary DES system based on pentaerythritol and choline chloride can

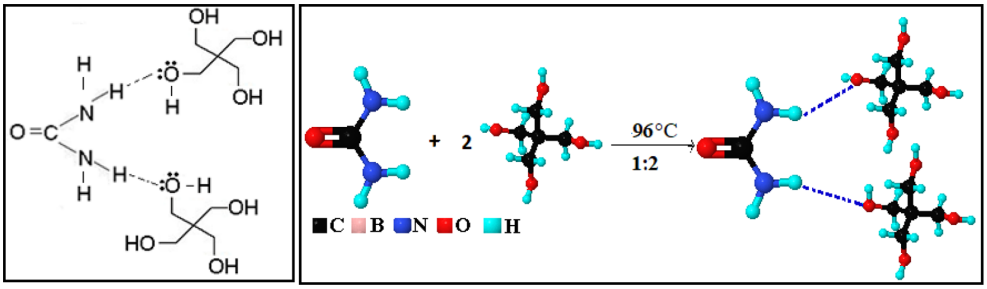

Fig. 4. Scheme of donor-acceptor interaction of urea- and pentaerythritol-based DES components

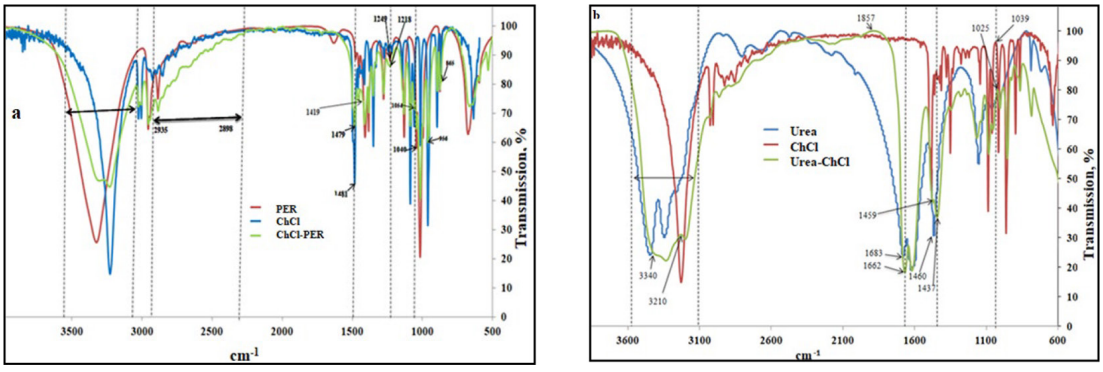

Fig. 5. IR spectra of: a – PER, ChCl and DES based on PER–ChCl; b – IR spectra of Urea, ChCl and DES based on Urea-ChCl

show obvious peaks at 3315, 2935, 1479, 1419, 1054, 956, 865 cm^{-1}. The peak at 3315 cm^{-1} belongs to the O-H stretching vibration.

The peak at 2935 cm^{-1} belongs to the C-H stretching vibrations. In addition, the peaks at 1479, 1085 and 956 cm^{-1} belong to CH_2 corner, CH_2CH_2-O stretching and C–C stretching vibrations in choline chloride, respectively. Also, for the binary DES system based on pentaerythritol and choline chloride, the broadening of the absorption band in the region from 3550 to 3100 cm^{-1} is observed due to the broadening of the absorption band in choline chloride.

Choline chloride groups belong to quaternary amide groups, and therefore in the initial state in the region from 3250 to 3207 cm^{-1} give one narrow absorption band. After the formation of hydrogen bonds between the DES molecules, this absorption band broadens from 3500 to 3100cm^{-1}. At the same time, for the binary DES system based on pentaerythritol and choline chloride, a shift in the absorption band is observed (Fig. 5a).

Peaks at 3340, 3210, 1857, 1662, 1437 and 1025 cm^{-1} are observed for the binary DES system based on urea and choline chloride. The peaks at 3340, 3210, 1617 cm^{-1} correspond to O-H, N-H and N-H stretching. The peaks displaying stretching vibrations of all redshifts in urea and choline chloride based DESs are in the range of 1662 to 1683 cm^{-1}, 1437 to 1460 cm^{-1} and 1025 to 1039 cm^{-1}, indicating that a strong hydrogen bond has formed between the components of the DESs [23–24]. Such red shifts have already been described in previous works. Fig. 6 shows that in the initial state urea has two absorption bands, choline chloride – one narrow absorption band, and after the formation of DES, the absorption band in urea widens to one band, and in choline chloride – increases. Also the broadening of the absorption band of DES based on urea and choline chloride in the region from 3600 to 3100 cm^{-1} indicates the formation of a large number of hydrogen bonds between DES molecules (Fig. 5a). This will lead to the formation of molecular complexes (Fig. 6 and 7).

Fig. 6 shows the mechanism of donor-acceptor interaction between pentaerythritol-based DESs and choline chloride, which results in the formation of a molecular complex in which choline chloride is the hydrogen bond acceptor and PER is the hydrogen bond donor (Fig. 7).

The results show that a large number of hydrogen bonds are present in DESs based on urea and choline chloride. This indicates that due to the donor-acceptor interaction of urea and choline chloride

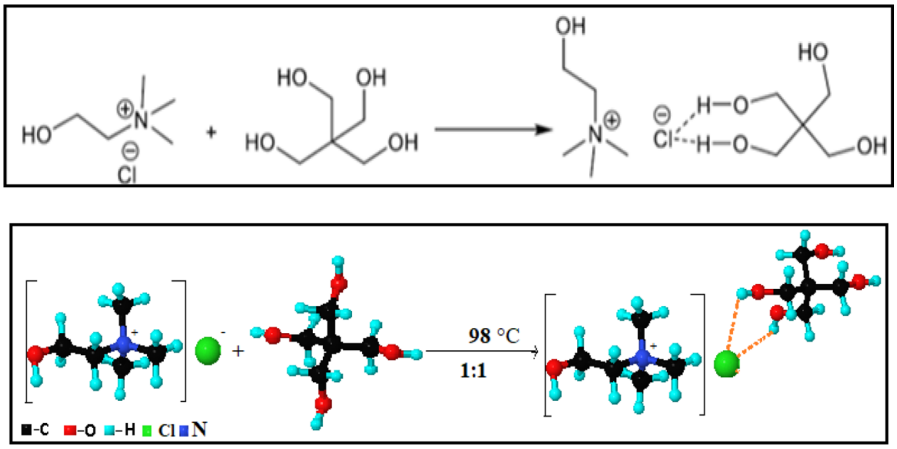

Fig. 6. Scheme of donor-acceptor interaction of DES components based on pentaerythritol and choline chloride

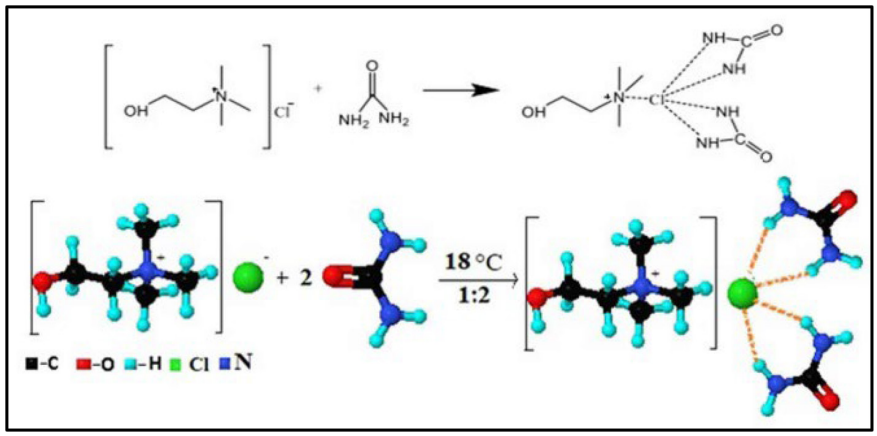

Fig. 7. Scheme of donor-acceptor interaction of urea and choline chloride based DES components

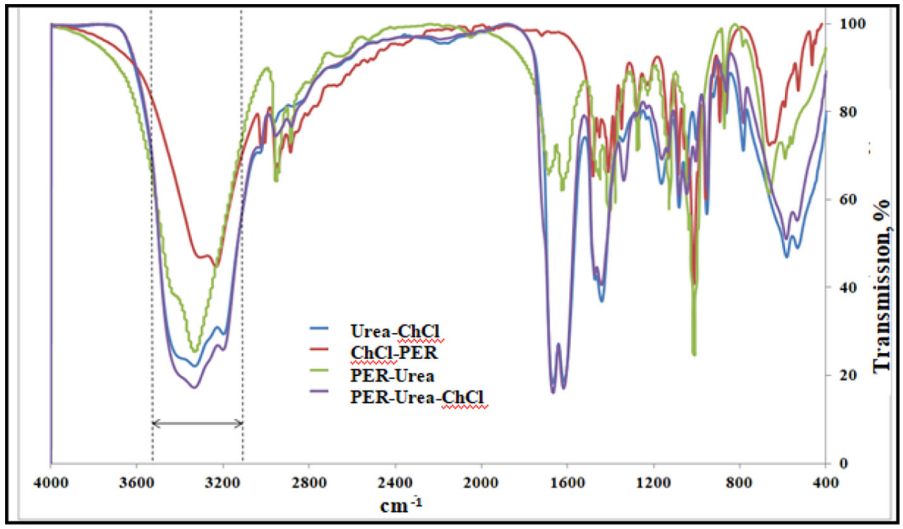

Fig. 8. IR spectra: Urea-ChCl; ChCl-PER; PER-Urea; PER-Urea-ChCl

based DES components, a molecular complex with low solidification temperature is formed (Fig. 7). In DESs based on urea and choline chloride, the latter is an acceptor and urea is a donor of hydrogen bonds.

Fig. 8 shows the IR spectrum of the ternary DES system based on pentaerythritol, urea and choline chloride.

In the IR spectrum of DES based on the ternary system "pentaerythritol – urea – choline chloride" peaks similar to those in the double systems are observed. Fig. 8 shows that the more components in the DES, the more the absorption peak widens in the region from 3600 to 3100 cm^{-1}. This indicates that a large number of strong hydrogen bonds (O–-H-O, O–-H-L, etc.) were formed in DES based on the ternary system "pentaerythritol – urea – choline chloride". At the same time, hydrogen bonds will lead to a decrease in the stability of the crystal lattice between the DES components. Consequently, even a small thermal energy is sufficient to overcome the lattice energy and destroy the solid crystal structure

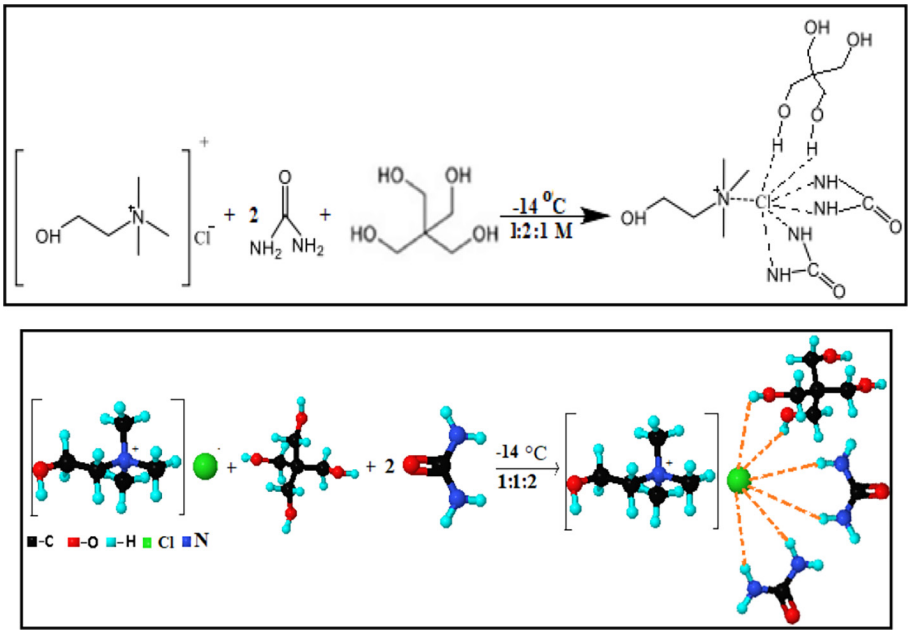

Fig. 9. Scheme of donor-acceptor interaction of DES components based on pentaerythritol, urea and choline chloride

of the components. This will result in a lower melting/crystallization temperature in the eutectic DES composition compared to the original components. The ternary DES system based on pentaerythritol, urea and choline chloride is a witness to this. As follows from IR spectra in the ternary system of DES based on pentaerythritol, urea and choline chloride there is a large number of hydrogen bonds. In this system, choline chloride acts as an acceptor of hydrogen bonds in relation to the donors – urea and pentaerythritol.

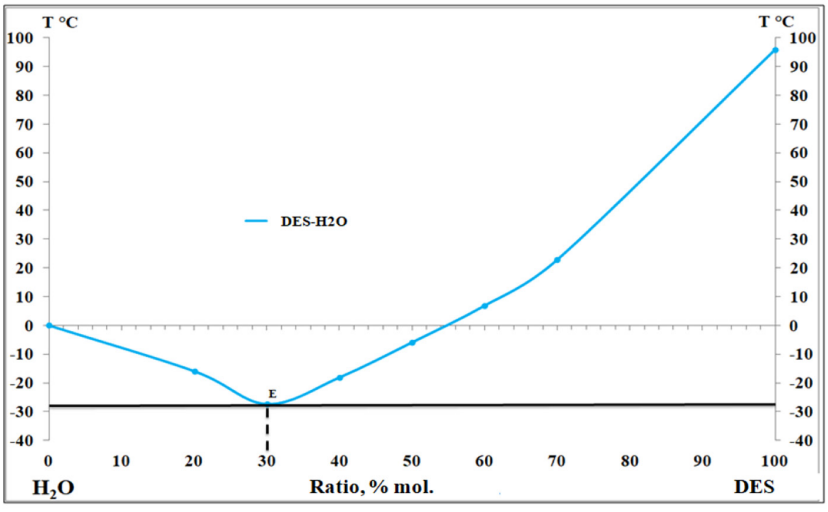

Fig. 10. Phase equilibrium diagram of the ternary system based on DES "pentaerythritol – urea – choline chloride" and water

Fig. 9 shows the donor-acceptor interaction mechanism of the DES components based on pentaerythritol, urea and choline chloride.

As a result of the phase equilibrium diagram study based on the ternary system of DES and water, it is shown that depending on the concentration of the ternary system of DES based on pentaerythritol, urea and choline chloride in aqueous solution, the solidification temperature decreases to 28.5 °C below zero (Fig. 10).

The obtained DES system "pentaerythritol – urea – choline chloride" can be the basis for chemical oil displacing composition with low pour point, the creation of which will be the next step of research.

Conclusion

Binary and ternary DES systems "pentaerythritol – choline chloride", "pentaerythritol –urea", "choline chloride – urea" and "pentaerythritol – urea – choline chloride" were synthesised and the coordinates of eutectic points were determined. The results of the study of IR spectra of binary and ternary systems of DESs based on pentaerythritol, urea and choline chloride showed that in all DESs there are a large number of hydrogen bonds. These bonds lead to a decrease in the melting/crystallization temperature in DES systems. The decrease in the melting/crystallization temperature of DES is explained by intermolecular forces and self-association in donor-acceptor interactions between components, which leads to a decrease in the lattice energy between DES molecules with the destruction of solid crystal structures of DES components. As a result, less energy is required to melt molecules than to melt individual components. By changing the structure and ratio of components in DESs, they can change their properties such as viscosity, density, melting point, hydrophobicity, etc. Consequently, their properties can be easily adapted and customized for different purposes.

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