STUDY OF THE POLYTHERMIC SOLUBILITY OF THE $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O SYSTEM

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ABSTRACT

In this work, the interaction of the components in the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system was studied by visual polythermal method in the temperature range from -42°C to 13°C. A polythermal solubility diagram of the system was constructed and the crystallization areas of ice, acetamipride, urea, and ureanitrate monoethanolammonium were separated in the diagram. The binary and ternary points of the separated phase lines on the solubility diagram, projections were drawn and examined. The [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] solution was selected considering the poor solubility of pure Acetamiprid in water. The [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O binary system was studied and the formed phases were analyzed using NMR spectroscopy (H^1 spectrum, C^{13} spectrum). In addition, ureanitrate monoethanolammonium compound was synthesized and analysed using physical chemical analysis methods (IR, Thermal).Dermatographic studies of the ureanitrate monoethanolammonium sample were studied in the mass size Δm_o = 45 mg, at a temperature change rate of 10°C min^{-1}, in the range from room temperature to ΔT = 800°C. The TG curves of the sample were determined by the results obtained from the dervatographic study.

Keywords: *insecticide, physiologically active substance, solubility diagram, polytherm, crystallization temperature, ureanitrate, ureanitrate monoethanolammonium, acetamiprid, X-ray phase analysis, NMR spectroscopy.*

INTRODUCTION

Water scarcity in agriculture directly affects nutrient dynamics [1], causing stress in plants and adversely affecting plant development, especially in plants growing in arid and semi-arid climates, this effect is noticeable [2].

Scientists' research shows that 28 - 38 % of the current cultivated areas have a problem of water shortage. This means that there is not enough moisture in the soil for the growth and development of agricultural crops, and there are no opportunities for irrigation of surface or underground water to eliminate these deficiencies [3]. The rational use of nutrients increases the resistance of plants to drought [4].

Fertilizers containing a large amount of nitrogen, such as urea, are regularly used in agriculture to increase productivity [5 - 7]. This substance dissolves well in water, is well absorbed by plants, does not accumulate in the soil as a residue, and strengthens plant immunity [8, 9]. It is also produced in plants as a breakdown product of arginine (Arg) and participates in important processes such as nitrogen reabsorption during seed germination and ripening. [10, 11].

Today, there are two main challenges facing farmers around the world. These include increasing the efficiency of using nitrogen fertilizers and eliminating the decrease

in the amount of organic matter in the soil [12].

Ethanolamines perform some important functions in plants, such as growth and development, stimulation, and effective synergism [13 - 16]. Under the influence of ethanolamine salts, ethylene is released in the plant and accumulates in the plant band [17], because of which the plant leaves fall prematurely, which provides the opportunity to harvest cotton with good quality [18].

Ethylene and other plant growth regulators are important in agricultural production. Plant growth regulators are now routinely used on a variety of crops worldwide. Ethylene, which is a plant growth hormone, has a strong effect on plants at almost all stages of development, from germination to fruit ripening and aging. In addition, because of research, its decisive role in the post-harvest physiology of agricultural products has been well studied [19].

The presence of ethylene, urea and nitrate groups in $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ affects the vegetative development of plants and shows that it is possible to control plant growth and aging depending on the ratio of components. This makes it possible to use this drug as a physiologically active substance.

Acetamiprid is one of the effective insecticides against sucking insects and is an odorless neonicotinoid synthetic organic compound. Under the influence of acetamiprid, the nervous system of insects becomes paralyzed [20, 21]. It is used in agriculture to protect plants from sucking insects such as aphids [22 - 24].

Considering the above, we were interested in studying the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system to obtain physiologically active substances with insecticidal properties.

Accordingly, the aim of the research is to develop the composition of multifunctional preparations based on ureanitrate, monoethanolamine and acetamiprid, which have physiologically active and insecticidal properties at the same time.

To study the interaction of urea nitrate, monoethanolamine and acetamiprid, which are the objects of research, and to physicochemical justify the production process of drugs with simultaneously physiologically active and insecticidal effect, binary and ternary systems was studied for the first time, analysed using physicochemical methods, and scientific information about the solubility of components was obtained.

EXPERIMENTAL

In the experiment, a TN-6 glass mercury thermometer with a detection limit of - 30 to 70°C and a TL-15 alcohol glass thermometer with a detection limit of - 100 to 20°C were used in the visual polythermal method [25]. For research purposes (pure for analysis) urea GOST 6671-77, "chemically pure" monoethanolamine (TU 2423-159-00203335-2004), nitrate acid (chemical pure for analysis) GOST 4461-77 were used.

Acetamiprid, reagent grade, under the name (E) - N1 - [(6-chloro-3-pyridyl)methyl] - N2-cyano-N1-methylacetamidine according to the IUPAC nomenclature was used [26].

400 MHz Nuclear Magnetic Resonance (JEOL JNM-ECZR, NMR) spectrometer was used for NMR analysis. X-Ray phase (Rigaku Mineflix 600, Japan) was used. Thermo Scientific TA Instruments STD 650 (TA Instruments Trios V5.1.1.46572.USA) for thermal analysis and A Perkin Elmer Spectrum IR (Version 10.7.2) was used to obtain IR analysis.

RESULTS AND DISCUSSION

Due to the poor solubility of acetamiprid in water, [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] solution was chosen [27].

A binary system consisting of [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] and water was studied from -1.9°C to the melting point of the initial components. In the diagram, the eutectic point corresponds to 1.8 % [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] and 98.2 % water at a temperature of -1.9°C (Fig. 1).

Samples were taken from different points of the phases formed in the diagram and analysed using NMR spectroscopy. It can be concluded from the analysis results of the studied binary system that in the binary system consisting of [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] and water, phase separation of ice and acetamiprid is observed (Fig. 2 and Fig. 3).

The urea nitrate (UN) was synthesized because of the reaction of nitric acid and urea under certain conditions [28]. The properties of UN are well revealed by the authors of the article [29, 30].

A vibration corresponding to (CO) and a vibration corresponding to (NO) at about 1300 cm^{-1} were observed at 1704.45 cm^{-1}. In the 2400 cm^{-1} region, there is a

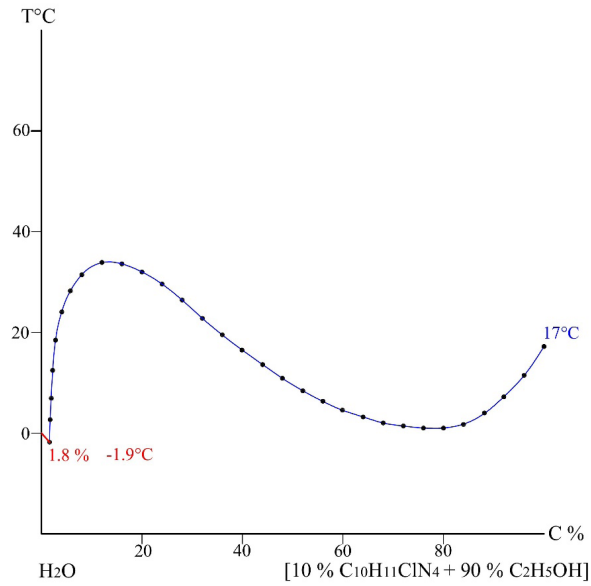

Fig. 1. Diagram of the binary system of [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] and water.

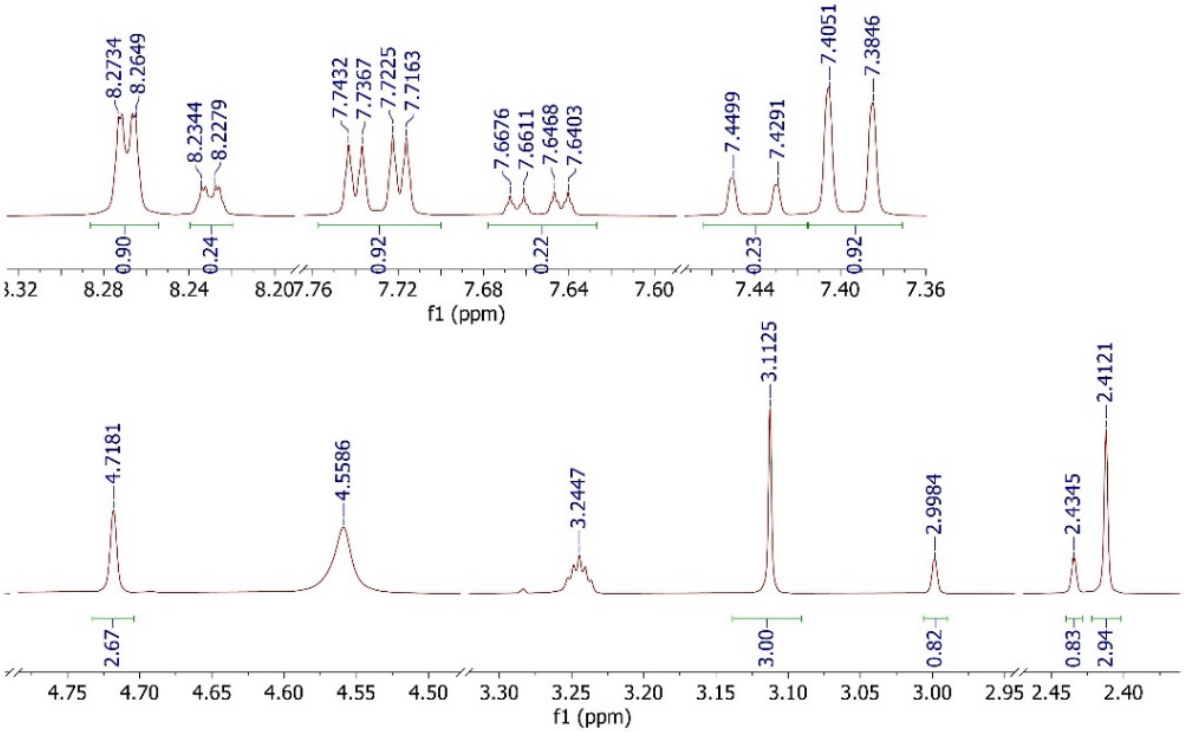

Fig. 2. NMR (H^1) spectrum.

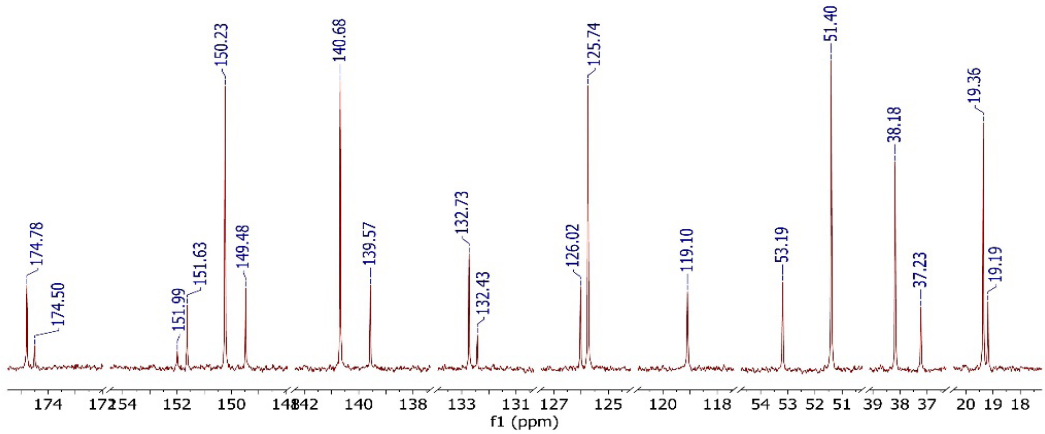

Fig. 3. NMR (C^{13}) spectrum.

characteristic vibration with a wide range between the hydrogen atom in the OH group and the oxygen atom bonded to the NO_2 group (C=O--H-ONO_2), this bond corresponds to a hydrogen bond. In addition, the asymmetric vibration related to NH_2 is in the region of 3404.52 cm^{-1}, 3350.45 cm^{-1} (NH stretch), 3197.99 cm^{-1} (NH_2 symmetric stretch), 1568.45 cm^{-1} (N-H), 1427.70 cm^{-1} (C-N), vibrations corresponding to 1243.84 cm^{-1} (NO_3^-) were observed.

MEA had bands around 3350 cm^{-1} (O-H) and 3288 cm^{-1} (N-H) and MEA had bonds in the high- and low-end of 2700 cm^{-1} (C-H stretch). These results were compared and confirmed with the literature [31, 32].

In the composition of ureanitrate monoethanol-ammonium (UNM) that authors offer [32], there is a bond at approximately 2900 cm^{-1} (C-H stretch), and a bond related to NO_3^- was observed around 1300 cm^{-1}. There is also a vibration at 1616 cm^{-1} corresponding to C=O. This allows us to justify the new UNM composition combination that we are offering.

A derivatographic study was conducted on the UNM sample with a mass size of $\Delta m_o = 45$ mg, at a temperature change rate of 10°C min^{-1}, from room temperature to DT = 800°C. Endothermic effects in the temperature range of 155.8 - 240.0°C and exothermic effects in the temperature range of 120.0 - 180.0; 201.1 - 262.3°C are observed in the DSC curves. TG curves show the change in mass of the UNM sample in the temperature range from 66.0°C to 517.0°C.

In addition, the results obtained from the dervatographic study determine the TG curves (red line) of the UNM sample. TG curves UNM show the state of mass change in the temperature range of the sample from 66.0°C to 517.0°C.

A polythermal solubility diagram of the system $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O was constructed between -42°C dan 13°C temperature using eleven internal cuts and binary systems (Fig. 6).

Internal lines I-VI are directed from $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ side to the [10 % $C_{10}H_{11}ClN_4$ + 90% C_2H_5OH] side, and lines VI-XI are directed to the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ side from [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] side.

During the study of this system, the crystallization areas of ice, acetamiprid, urea and ureanitrate monoethanolammonium were separated. All phases in the diagram combine at one ternary point.

The ternary point corresponds to the temperature of -42°C, the composition of the liquid phase is 31.6 % UNM, 46.4 % acetamiprid and 22 % water, and this point is limited by the crystallization areas of urea, acetamiprid and water (Fig. 6 and Table 1).

During the construction of the polythermal

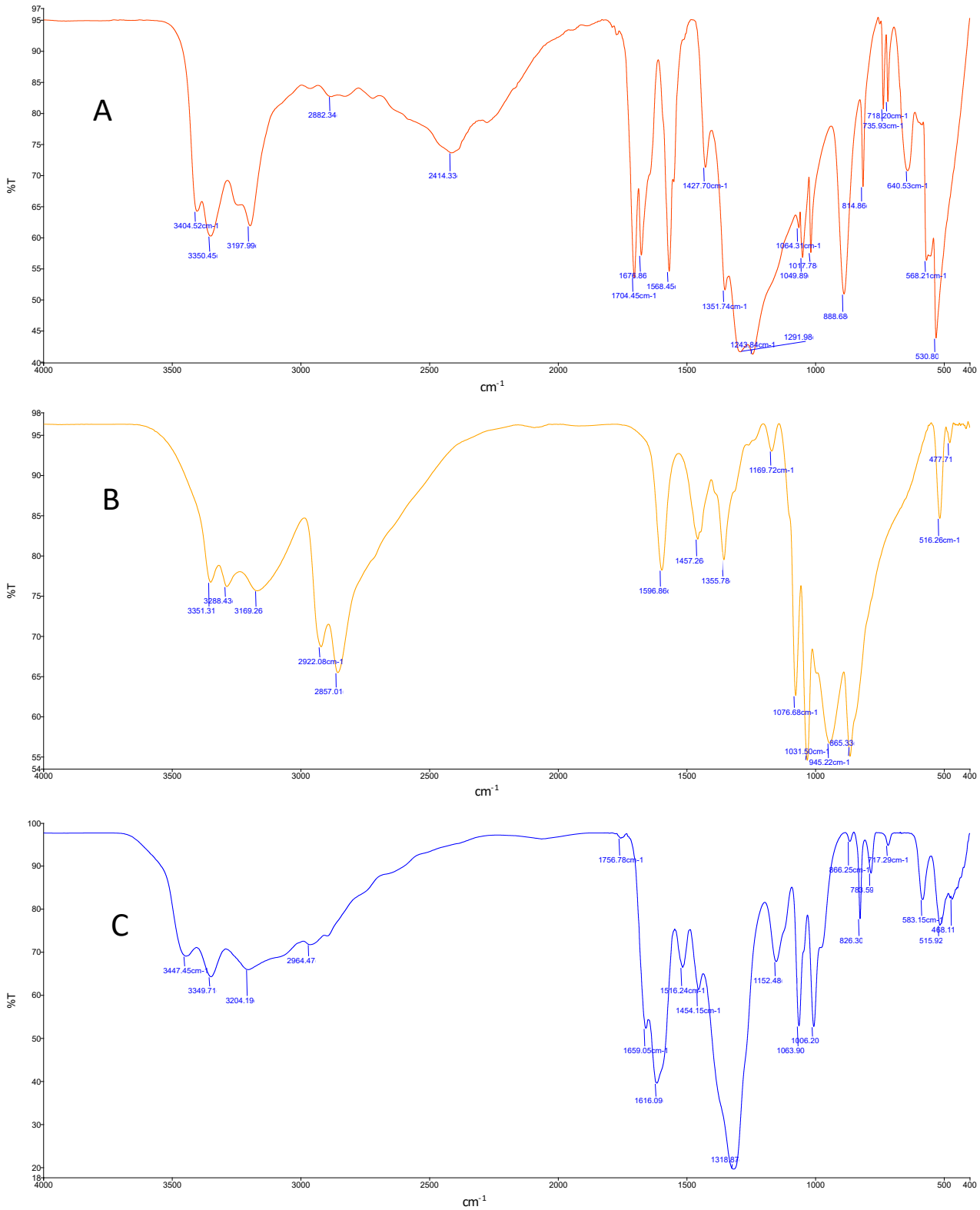

Fig. 4. IR spectra: A - UN; B -MEA (Monoethanolamine); C - UNM (ureanitrate monoethanolammonium).

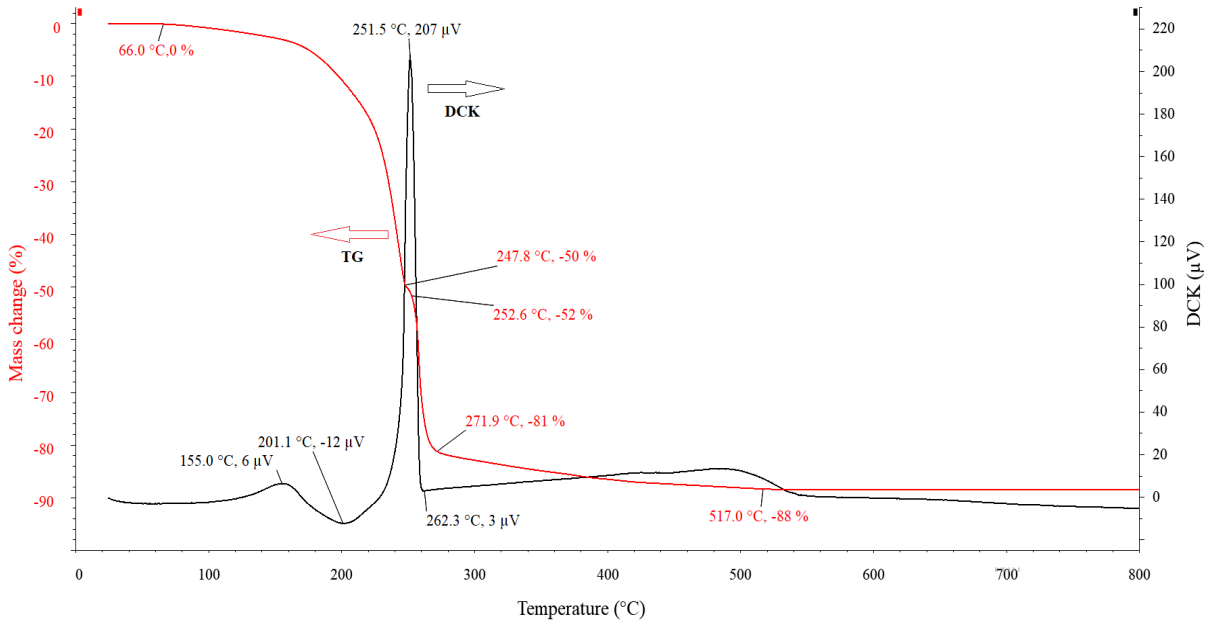

Fig. 5. TG (Thermogravimetric) and DSC (Differential Scanning Calorimeter) curves of UNM sample.

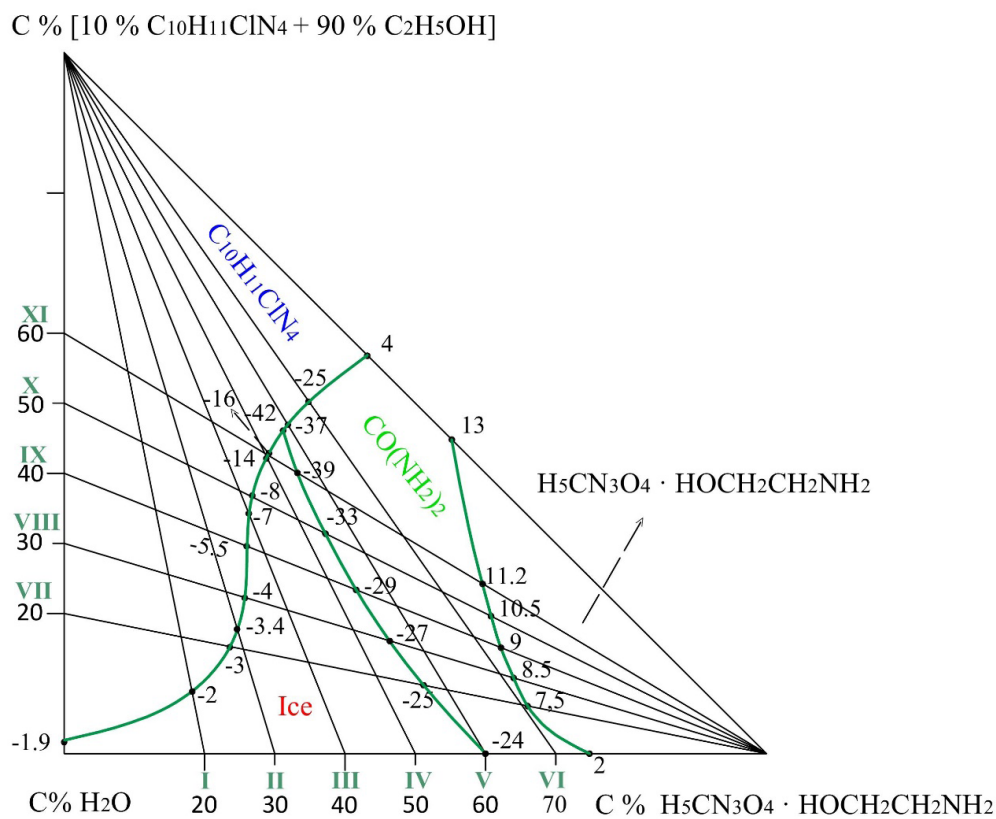

Fig. 6. Polythermal solubility diagram of the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system.

Table 1. Binary and ternary points of the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system.

Composition of the liquid phase, wt. %,			Cryst., T°C	Solid phase
$H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$	$C_{10}H_{11}ClN_4$	H_2O		
---	1.80	98.2	-1.90	Ice + $C_{10}H_{11}ClN_4$
18.4	9.20	72.4	-2.00	
24.0	15.2	60.8	-3.00	
25.6	22.0	52.4	-4.00	
26.0	29.6	44.4	-5.50	
26.4	35.0	38.6	-7.00	
27.8	36.5	35.7	-8.00	
29.0	41.6	29.4	-14.0	
29.2	42.4	28.5	-16.0	
31.6	46.4	22.0	-42.0	Ice + $C_{10}H_{11}ClN_4$ + $CO(NH_2)_2$
32.0	47.0	21.0	-37.0	$C_{10}H_{11}ClN_4$ + $CO(NH_2)_2$
34.8	50.0	15.2	-25.0	
43.0	57.0	---	4.00	
33.0	49.0	8.00	-39.0	Ice + $CO(NH_2)_2$
37.0	31.6	31.4	-33.0	
41.6	23.2	35.2	-29.0	
46.5	16.0	37.5	-27.0	
51.0	9.80	39.2	-25.0	
60.0	---	40.0	-24.0	
75.0	---	25.0	2.00	$H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ + $CO(NH_2)_2$
66.0	7.00	27.0	6.00	
64.0	10.8	25.2	7.50	
62.0	15.2	22.8	9.00	
60.8	19.6	19.6	10.5	
59.0	24.4	16.6	11.2	
55.0	45.0	---	13.0	

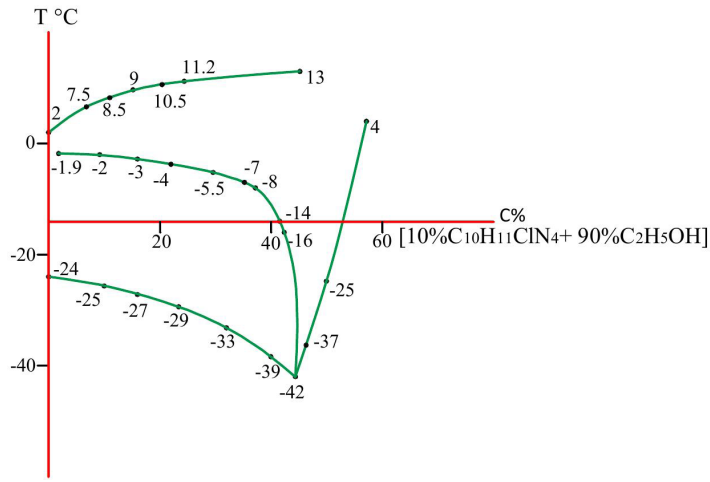

Fig. 7. Projection of the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system.

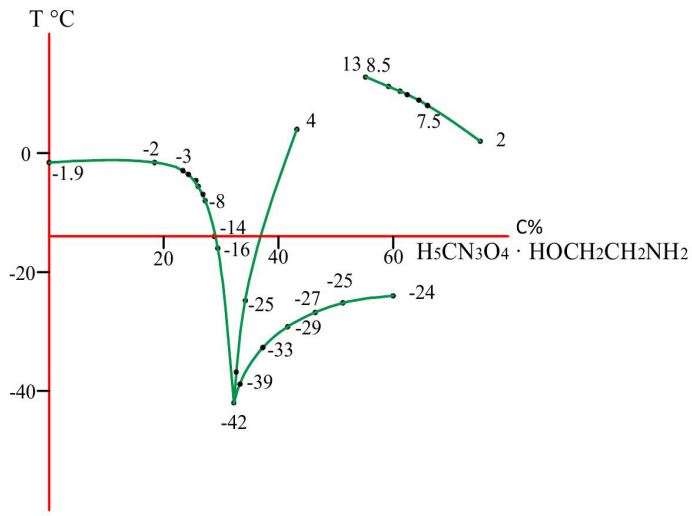

Fig. 8. Projection of the $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O system.

solubility diagram of $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] - H_2O, the projections of the polythermal solubility curves of this system were drawn and the correlation was recorded.

The initial projection was studied according to the direction of increasing concentration of [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] from the side of UNM (Fig. 7). The second projection was analysed according to the direction of increasing concentration of UNM from the [10 % $C_{10}H_{11}ClN_4$ + 90 % C_2H_5OH] side (Fig. 8).

CONCLUSIONS

This $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + + 90 % C_2H_5OH] - H_2O system was studied by the authors for the first time and scientific information about the interaction of components was obtained. A polythermal solubility diagram of the system was constructed and the crystallization areas of ice, acetamiprid, urea, and ureanitrate monoethanolammonium were separated in the diagram.

As a result of the analysis of the studied solubility system, it became known that it is possible to obtain a physiologically active drug with insecticidal properties based on $H_5CN_3O_4 \cdot HOCH_2CH_2NH_2$ - [10 % $C_{10}H_{11}ClN_4$ + + 90 % C_2H_5OH] - H_2O.

Authors' contributions: B.B., Z.Sh.: Study conception and design; B.B.: data collection; B.B., Z.Sh. and N.K.: analysis and interpretation of results; B.B., Z.Sh.: draft manuscript preparation. All authors reviewed the results and approved the final version of the manuscript. The B.B. confirms sole responsibility for the following: study conception and design, data collection, analysis and interpretation of results, and manuscript preparation.

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