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SOLUBILITY POLYTHERM IN THE SYSTEM Ca(ClO3)2·4CO(NH2)2-ClCH2COOH·(HOC2H4)3N-H2O -H2O

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ABSTRACT: Background. Cotton growing is one of the most important agricultural sectors in Uzbekistan. To ensure high-quality harvesting of raw cotton in a short time, defoliation is an agronomic measure. As a result of defoliant action, leaf fall improves air circulation among cotton rows, allowing sunlight to fall directly on the developing bolls. Therefore, special attention is paid to the production of low-toxicity, highly effective defoliants.

Aim of the Study. The aim of the study is to physicochemically substantiate the process of producing an effective defoliant based on calcium tetracarbamidochlorate and triethanolamine chloroacetate.

Methodology. To physicochemically substantiate the process for producing a complex cotton defoliant, data were obtained on the solubility and rheological properties of solutions in systems containing calcium tetracarbamidochlorate and triethanolamine chloroacetate. Visual-polythermal and pycnometric methods were used.

Scientific novelty. For the first time, the solubility of a system consisting of calcium tetracarbamidochlorate, triethanolamine monochloroacetate, and water was studied. Based on binary systems and internal sections of polytherms, a polythermal solubility diagram was constructed for the Ca(ClO3)2·4CO(NH2)2 - ClCH2COOH·(HOC2H4)3N-H2O system in the temperature range from -25.0 to 65.0°C. The phase diagram delineates the crystallization fields of the starting materials and the new compound CH2ClCOOH·Ca(ClO3)2·(C2H4OH)3N.

Results obtained. In the studied system, the crystallization fields of ice, Ca(ClO3)2·4CO(NH2)2·2H2O, ClCH2COOH·(HOC2H4)3N, and the new compound CH2ClCOOH·Ca(ClO3)2·(C2H4OH)3N were delineated.

Keywords: calcium tetracarbamidochlorate, triethanolamine monochloroacetate, solubility diagram.

Features:

- crystallization fields of the components have been delineated into systems;

- the formation of a new compound, CH2ClCOOH Ca(ClO3)2 (C2H4OH)3N, has been established;

- optimal process parameters for producing a liquid defoliant have been established.

INTRODUCTION

Calcium chlorate-containing, mild- and complex-action defoliants with physiologically active substances are currently used to obtain high yields of high-quality cotton. One of the most promising, agrochemical, and economically feasible methods for increasing the effectiveness of defoliants, increasing crop yields, and improving the quality of agricultural products is the combined use of defoliants with ethylene producers and mineral fertilizers [1-3]. There is insufficient data in the literature to substantiate the physicochemical principles and technology for producing complex-action defoliants based on calcium tetracarbamide chlorate, which contain physiologically active substances and nutrients. Calcium tetracarbamidochlorate is of significant interest in the synthesis of new effective defoliants. It is a chlorate-containing defoliant synthesized from calcium chlorate and urea, which are produced domestically [4]. Triethanolamine monochloroacetate, a chemical intermediate, is also of interest.

The aim of this study is to physicochemically substantiate the process for producing an effective defoliant based on calcium tetracarbamidochlorate and triethanolamine chloroacetate.

RESEARCH OBJECT AND METHODS

The objects of study were calcium tetracarbamidochlorate and triethanolamine monochloroacetate. Triethanolamine chloroacetate forms colorless crystals. Calcium tetracarbamidochlorate was obtained by smelting urea with calcium chlorate at a molar ratio of 4:1 (4CO(NH2)2:Ca(ClO3)2) [5]. For quantitative chemical analysis, generally accepted methods of analytical chemistry were used, in particular, the amount of chlorate ion was determined by volumetric permanganometric methods [6], and calcium by volumetric complexometric methods [7]. The content of elemental carbon and hydrogen was determined according to the method [8-10]. To study the solubility of the components, a visual-polythermal method was used [11] using a TN-6 mercury-in-glass thermometer with a measurement range from -30 to +60°C and a TL-15 mercury-in-glass thermometer with a measurement range from -100 to +20°C. [12]. IR absorption spectra of the starting components and the compounds under study were recorded in the frequency range of 4000 - 400 cm–1 on a SHIMADZU MIRacle10 FT-IR [13-14]. RESULTS AND DISCUSSION

The binary system ClCH2COOH·(HOC2H4)3N -H2O was studied in the temperature range from -5.9°C to 70.0°C. The polythermal solubility diagram is characterized by the presence of ice crystallization branches and the monochloroacetic acid amine salt at a temperature of -5.9°C and a concentration of 31.0% ClCH2COOH·(HOC2H4)3N and 69.0% H2O. The obtained results are consistent with the literature data presented in [15-18].

The binary system Ca(ClO3)2·4CO(NH2)2–H2O is characterized by ice crystallization branches and calcium tetracarbamidochlorate with a transition point at -18.0°C, in which the concentration of Ca(ClO3)2·4CO(NH2)2 is 52.0%. The results are in good agreement with literature data [5].

For the first time, the solubility of the Ca(ClO3)2·4CO(NH2)2 - ClCH2COOH·(HOC2H4)3N - H2O systems was studied visually using a polythermal method in the temperature range from -25.0°C to 65.0°C. A solubility diagram was constructed using binary systems and a series of internal sections. Sections I-III are drawn from the ClCH2COOH·(HOC2H4)3N -H2O side to the apex of Ca(ClO3)2·4CO(NH2)2, and sections IV-VI are drawn from the calcium tetracarbamide chlorate - water side to the pole of ClCH2COOH·(HOC2H4)3N. In the polythermal diagram of the solubility of the systems, the crystallization fields of ice, Ca(ClO3)2·4CO(NH2)2·2H2O, ClCH2COOH·(HOC2H4)3N and the new compound CH2ClCOOH·Ca(ClO3)2·(C2H4OH)3N are highlighted. These fields converge at two triple nodal points of the system, for which the equilibrium solution compositions and the corresponding crystallization temperatures are determined (Fig. 1, Table 1).

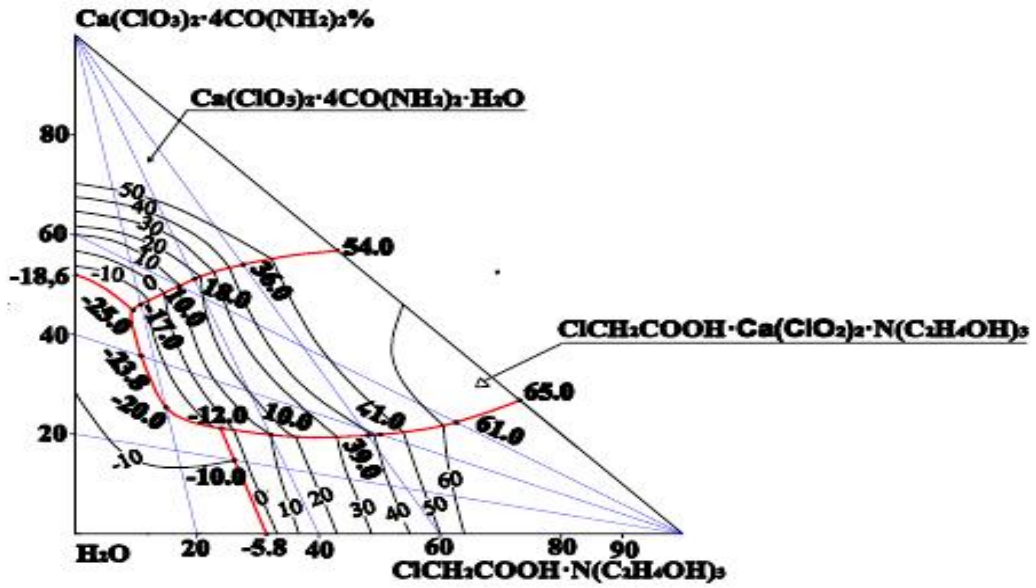

Fig. 1. Polythermal solubility diagram of the system

Ca(ClO3)2·4CO(NH2)2- ClCH2COOH·(HOC2H4)3N -H2O

The polythermal state diagram of the system shows solubility isotherms at temperatures of -30, -20, -10, 0, 10, 20, 30, and 40°C.

Table 1. Double and triple points of the system

$Ca(ClO_3)_2 \cdot 4CO(NH_2)_2$- $CH_2ClCOOH \cdot Ca(ClO_3)_2 \cdot (C_2H_4OH)_3N$ -H_2O

Состав жидкой фазы, %	К р .	Т в е р д а я ф а з

$Ca(ClO_3)_2 \cdot 4CO(NH_2)_2$	$CH_2ClCOOH \cdot (C_2H_4OH)_3 N$	H_2O		
1	2	3	4	5
52.0	-	48.0	-18.6	Лед + $Ca(ClO_3)_2 \cdot 4CO(NH_2)_2$
44.8	9.6	45.6	-25.0	Лед + $Ca(ClO_3)_2 \cdot 4CO(NH_2)_2$ + $ClCH_2COOH \cdot Ca(ClO_3)_2 \cdot (C_2H_4OH)_3N$
46.0	11.0	43.0	-17.0	тожи
49.8	17.0	33.2	10.0	-//-
51.4	19.5	29.1	18.0	-//-
54.2	27.5	18.3	36.0	-//-
56.4	43.4	0.2	54.0	-//-
36.5	10.8	52.7	-23.8	Лед + $ClCH_2COOH \cdot Ca(ClO_3)_2 \cdot (C_2H_4OH)_3N$
25.4	15.	59.4	-20.0	-//-
21.2	24.0	54.8	-12.0	Лед + $ClCH_2COOH \cdot Ca(ClO_3)_2 \cdot (C_2H_4OH)_3N$ + $ClCH_2COOH \cdot (C_2H_4OH)_3N$
14.5	26.4	59.1	-10.0	Лед + $ClCH_2COOH \cdot (C_2H_4OH)_3N$
-	31.0	69.0	-5.9	Лед + $ClCH_2COOH \cdot (C_2H_4OH)_3N$
19.6	32.2	48.2	10.0	$ClCH_2COOH \cdot Ca(ClO_3)_2 \cdot (C_2H_4OH)_3N$ + $ClCH_2COOH \cdot (C_2H_4OH)_3N$
19.9	48.0	32.1	39.0	-//-
21.2	49.9	28.9	41.0	-//-
23.0	61.2	15.8	61.0	-//-
26.9	73.0	0.1	65.0	-//-

To identify the new compound, we isolated it in crystalline form from the proposed crystallization region and identified it using chemical and physicochemical analysis.

The chemical composition of the solid phase isolated from the proposed crystallization region of ClCH2COOH Ca(ClO3)2 (C2H4OH)3N corresponds to the following results:

Found, %: Ca2+ -9.11; ClO3- -35.3; N -3.81.

Calculated, %: ClCH2COOH Ca(ClO3)2 (C2H4OH)3N, %: Ca2+ -8.87; ClO3- -37.06; N -3.10.

The solubility of the obtained substance in water (in %) is 11.76 at 10°C, 17.62 at 20°C, 21.62 at 30°C, 27.02 at 40°C, and 33.81 at 50°C. It is insoluble in organic solvents—alcohol, toluene, benzene, acetone, and chloroform.

IR spectroscopic studies show that the spectra of CH2ClCOOH·(C2H4OH)3N and the new compound ClCH2COOH·Ca(ClO3)2·(C2H4OH)3N contain all the characteristic stretching and deformation vibration bands (Fig. 2).

The IR spectrum of CH2ClCOOH·(C2H4OH)3N is based on the stretching vibrations of the OH group linked by hydrogen bonds (Fig. 2). The bands at 2938 and 2863 cm-1 correspond to νas (CH2) and νs (CH2). The absorption lines observed at 1645, 1075 and 1040 cm-1 correspond to the experimentally determined δ(OH), ν (CH) and ν (C-O) groups, respectively. In the spectrum of CH2ClCOOH•(C2H4OH)3N, the OH group was introduced into line 1 of the 3-N spectrum, while the frequency band at 3255 cm-1 was based on the symmetric stretching vibrations of this group. The lines of antisymmetric and symmetric stretching vibrations of the group were located at 3140 and 3000 cm-1. The observed vibrations in the 1380 and 1310 cm-1 frequency range are reported to be associated with antisymmetric and symmetric stretching vibrations of the ionized carboxyl group of monochloroacetic acid, while the data at 1150, 100-980, and 550 cm-1, as well as the absorption lines, remained unchanged compared to the spectrum of triethanolamine.

In IR spectroscopic analysis of the compound, the stretching vibrations of the OH group of monochloroacetic triethanolamine disappeared. The vibration frequencies of as(ClO3) and as(ClO3) are observed at 974 and 914 cm-1 (Fig. 4.20-2). This suggests that the OH group of monochloroacetic triethanolamine is replaced by ClO3- ions. In the new compound, the 2360 cm-1 bands correspond to the stretching vibrations of CH2 bonds, while the 1124 cm-1 bands correspond to the stretching vibrations of N-H bonds. The range from 617 to 499 cm-1 belongs to the asymmetric and symmetric stretching vibrations of the COOH group. Thus, the stretching vibrations of the OH lines disappeared from the absorption spectra of the compound, while vibrations of the as(ClO3) and as(ClO3) ions were observed.

The IR spectrum of CH2ClCOOH·(C2H4OH)3N is characterized by absorption bands at 667.4 and 763.81 cm-1, due to antisymmetric and symmetric stretching vibrations of the C-Cl group. The bands at 929.69 cm-1 are caused by the out-of-plane, deformation vibration of the OH bond of the carboxyl group, and the stretching vibrations as(C=O) and s(C=O) of the carboxyl group are assigned to absorption bands in the frequency range of 1593.2, 1396.46 and 1247.94 cm-1.

The IR spectra of ClCH2COOH Ca(ClO3)2 (C2H4OH)3N differ from those of free monochloroacetic acid sodium salt by the presence of new absorption bands in the frequency range of 3363.86 and 3537.45 cm-1. These are characteristic of antisymmetric and symmetric stretching vibrations of OH groups, which are associated with the formation of hydrogen bonds in crystallized water.

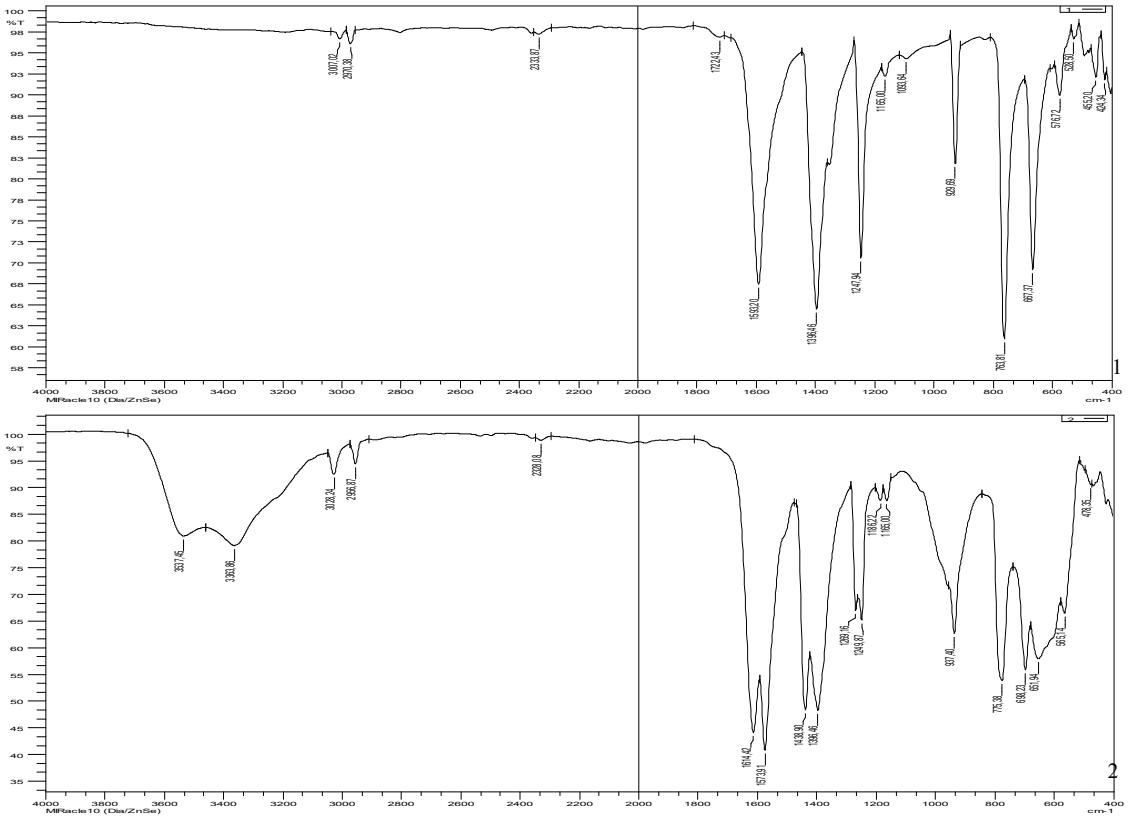

Fig. 2. IR spectra of triethanolamine monochloroacetate (1) and the compound ClCH2COOH Ca(ClO3)2 (C2H4OH)3N (2).

CONCLUSION

Thus, by studying the solubility diagram of the Ca(ClO3)2·4CO(NH2)2–CH2ClCOOH (C2H4OH)3N-H2O system, we established the formation of a new compound, ClCH2COOH Ca(ClO3)2 (C2H4OH)3N. This formation was confirmed by chemical and physicochemical analysis.

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