

## OBTAINING A NEW DEFOLATING COMPOSITION WITH PHYSIOLOGICAL ACTIVITY

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**Abstract:** This article presents research on the development of a basic technological scheme for obtaining a defoliant with physiologically active additives in order to improve the quality. Studied the composition-properties of the diagram with the participation of chlorates and chlorides of calcium and magnesium, carbamide, ethanol and monoethanolammonium acetate. Revealed optimal rheological and agrochemical properties of solutions. A process flow diagram has been developed for obtaining a new complex acting defoliant.

**Keywords:** defoliants, physiologically active substances, chlorates and chlorides of calcium, magnesium, calcium-magnesium chlorate defoliant, carbamide, ethanol, monoethanolamine acetate

### Introduction

In order to efficiently and timely harvest raw cotton, it is extremely necessary to carry out pre-harvest removal of cotton leaves using chemical preparations, the so-called defoliants. Defoliation is an important agronomic measure, without which it is impossible to achieve early harvest of raw cotton.

Currently, drugs for protecting crops, defoliants and plant growth regulators are mainly brought from abroad in the form of active principles and preparative forms ... For the production of chlorate magnesium defoliant (contains 36% of the active substance) [1] at JSC "Ferganaazot" the original raw material bischofite (magnesium chloride) is imported from Volgograd (Russia) or Turkmenistan for foreign currency. This increases the cost of the defoliant.

To solve this problem, employees of IONKh AN RUZ and FerPI have developed a technology for obtaining a new chlorate calcium-magnesium defoliant by using as a raw material, instead of imported "bischofite", products of hydrochloric acid decomposition of dolomites m. "Shorsu" and "Pachkamar" [2,3].

The purpose of this work is to develop a technology for obtaining new defoliants of complex action, having defoliating activity, accelerating the maturation and opening of bolls based on a calcium-magnesium chlorate preparation obtained from dolomites from the Navbahor deposit [4].

### Objects and methods of research

The objects of research are calcium chlorate and magnesium chlorate.  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{H}_2\text{O}$  was obtained on the basis of the exchange reaction of fused calcium chloride with sodium chlorate in an acetone medium as described in [5]. As a result of the exchange reaction, a solution of calcium chlorate in acetone was obtained. After separation of the acetone extract from the solid phase and removal of acetone under vacuum at a temperature of 30–35 ° C, a white crystalline product was isolated from the resulting thick mass by cooling, which was purified by recrystallization.  $\text{Mg}(\text{ClO}_3)_2 \cdot 6\text{H}_2\text{O}$  was synthesized by the method described in [6] and monoethanolammonium acetate was synthesized by reacting acetic acid with monoethanolamine taken at a molar ratio of 1: 1 and vigorous stirring.

the system was studied using the visual-polythermal method [7].

In quantitative chemical analysis, well-known methods of analytical chemistry were used, in particular: chlorate ion was determined by volumetric

permanganometric method [1]; calcium and magnesium were determined by the volumetric complexometric method [8]. The content of elemental nitrogen, carbon, hydrogen was carried out according to the method [9].

### Results and discussion

Previous studies established the optimal composition of the preparation, including calcium-magnesium chlorate defoliant, urea and ethanol: { [19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0 % C<sub>2</sub>H<sub>5</sub>OH}.

For the physicochemical substantiation and recommendation of the process of obtaining a complex defoliant with high defoliation activity, "soft"

action and accelerating the process of ripening and opening of cotton bolls based on calcium-magnesium chlorate preparation, urea, ethanol and the physiologically active substance monoethanolamine acetate, the physicochemical properties of solutions in the system {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0% C<sub>2</sub>H<sub>5</sub>OH} · CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH.

The crystallization temperature, pH, density and viscosity of solutions of the studied system were determined we are depending on the ratio of the components. On the basis of the data obtained (Table 1), the diagrams "composition-properties" of the system were constructed (Fig. 1).

Table 1: Dependence of the change in the physicochemical properties of solutions on the composition in the system {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45.17% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0% C<sub>2</sub>H<sub>5</sub>OH} · CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH

№	Content of components,%		t, °C	pH	η, mm <sup>2</sup> /c	d, g/sm <sup>3</sup>
	{[19,37%Ca(ClO <sub>3</sub> ) <sub>2</sub> + 15,06%Mg(ClO <sub>3</sub> ) <sub>2</sub> + 3,72%CaCl <sub>2</sub> +2,68%MgCl <sub>2</sub> + 45,17%H <sub>2</sub> O] +10,0%CO(NH <sub>2</sub> ) <sub>2</sub> +4,0%C <sub>2</sub> H <sub>5</sub> OH}	CH <sub>3</sub> COOH· NH <sub>2</sub> C <sub>2</sub> H <sub>4</sub> OH				
1	100	-	-2.3	4.50	8.94	1.4372
2	99.95	0,05	-2,1	4,53	8,96	1,4390
3	99.92	0,08	-1,9	4,55	8,97	1,4410
4	99.88	0,12	-1,7	4,57	8,99	1,4420
5	99.82	0,18	-1,4	4,62	9,03	1,4430
6	99.76	0,24	-0,9	4,67	9,06	1,4450
7	99.7	0,30	-0,3	4,73	9,09	1,4470
8	99.6	0,40	0,5	4,79	9,15	1,4490
9	99.55	0,45	1,2	4,85	9,18	1,4530
10	99.51	0,49	1,9	4,88	9,22	1,4540
11	99.44	0,56	3,0	4,94	9,25	1,4580
12	98.35	0.65	3,8	5,03	9,30	1,4600
13	99.28	0.72	5,2	5,14	9,35	1,4630
14	99.21	0.79	6,0	5,17	9,40	1,4650
15	99.12	0.88	7,4	5,26	9,47	1,4690
16	99.0	1.0	9,5	5,38	9,56	1,4740

Figure 1 shows that during the dissolution of monoethanolamine acetate in a solution of calcium-magnesium chlorate preparation containing 10% carbamide, 4.0% ethanol, the crystallization temperature, pH, viscosity and density of the newly formed solutions gradually increase. On the "composition-properties" curve of the diagram for the studied intervals, no sharp jumps and breaks are observed. This pattern is also preserved when studying the solubility of complex systems with the participation of the above components [10]. This is explained by the fact that within the studied limits of the components in the system {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45.17% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> +

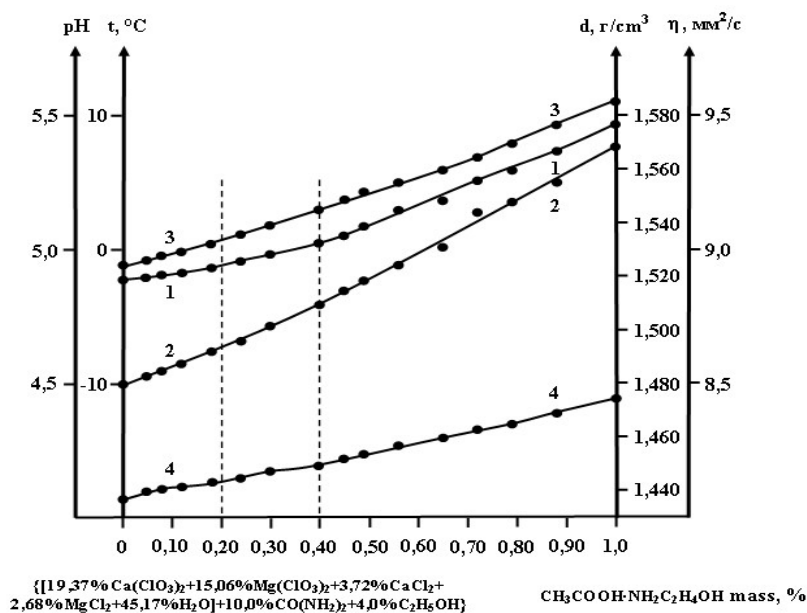


Fig. 1. The dependence of the change in the crystallization temperature (1),

pH (2), density (3) and viscosity (4) of solutions on the composition in the system {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45.17% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0% C<sub>2</sub>H<sub>5</sub>OH} - CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH

4.0% C<sub>2</sub>H<sub>5</sub>OH} -CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH there is no change in the crystallizing solid phases.

In order to select the optimal ratio of components in the defoliant, obtained on the basis of calcium-magnesium chlorate, carbamide, ethanol and monoethanolamine acetate, agrochemical tests of various defoliant compositions were carried out on cotton. The results showed that the composition of the defoliant in which the ratio of the components is {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45.17% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0% C<sub>2</sub>H<sub>5</sub>OH} and CH<sub>3</sub>COOH · NH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH equal to 1.0: 0.002 ÷ 0.004 has a high defoliation activity and a "mild" effect on cotton, and also has a positive effect on the acceleration of ripening and opening boxes.

Based on the results of studying the "composition-property" of this system, as well as agrochemical tests, it follows that to obtain a defoliant of complex action containing monoethanolamine acetate in its composition, it is necessary in a solution of the composition {[19.37% Ca (ClO<sub>3</sub>)<sub>2</sub> + 15.06% Mg (ClO<sub>3</sub>)<sub>2</sub> + 3.72% CaCl<sub>2</sub> + 2.68% MgCl<sub>2</sub> + 45.17% H<sub>2</sub>O] + 10.0% CO (NH<sub>2</sub>)<sub>2</sub> + 4.0% C<sub>2</sub>H<sub>5</sub>OH} dissolve CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH at a mass ratio of 1.0: 0.002 ÷ 0.004. The resulting defoliant solution has the following physicochemical properties: crystallization temperature 0 ÷ -2.50C, pH = 4.65 ÷ 4.79, viscosity 9.04 ÷ 9.15 mm<sup>2</sup> / s and density 1.4440 ÷ 1.4490 g / cm<sup>3</sup> and has the following composition, wt%: 34 ÷ 36% Ca (ClO<sub>3</sub>)<sub>2</sub> + Mg (ClO<sub>3</sub>)<sub>2</sub>; 10% CO (NH<sub>2</sub>)<sub>2</sub>; 4% C<sub>2</sub>H<sub>5</sub>OH; 0.2 ÷ 0.4% CH<sub>3</sub>COOH · NH<sub>2</sub>C<sub>2</sub>H<sub>4</sub>OH, the rest is water.

The production of the new defoliant is carried out in a batch mode using crystalline carbamide, monoethanolamine acetate, as well as a solution of calcium-magnesium chlorate, ethanol and monoethanolamine acetate. The essence of the method for the production of a new defoliant is the sequential dissolution of the calculated amounts of carbamide, ethanol and monoethanolamine acetate in a liquid calcium-magnesium chlorate preparation.

Production of the preparation consists of the following main stages:

- loading the calcium chlorate solution into the reactor -magnesium preparation;
- loading and dissolving carbamide in a solution of calcium-magnesium chlorate preparation and obtaining a homogeneous solution;
- loading ethanol, monoethanolamine acetate into the mixing reactor and dissolving the latter in ethanol; - loading into the synthesis reactor of a solution consisting of a calcium-magnesium preparation chlorate, carbamide and a solution of ethylene producers, dissolving them to obtain a complex defoliant;
- packing the resulting product.

Figure 2 shows block scheme of obtaining

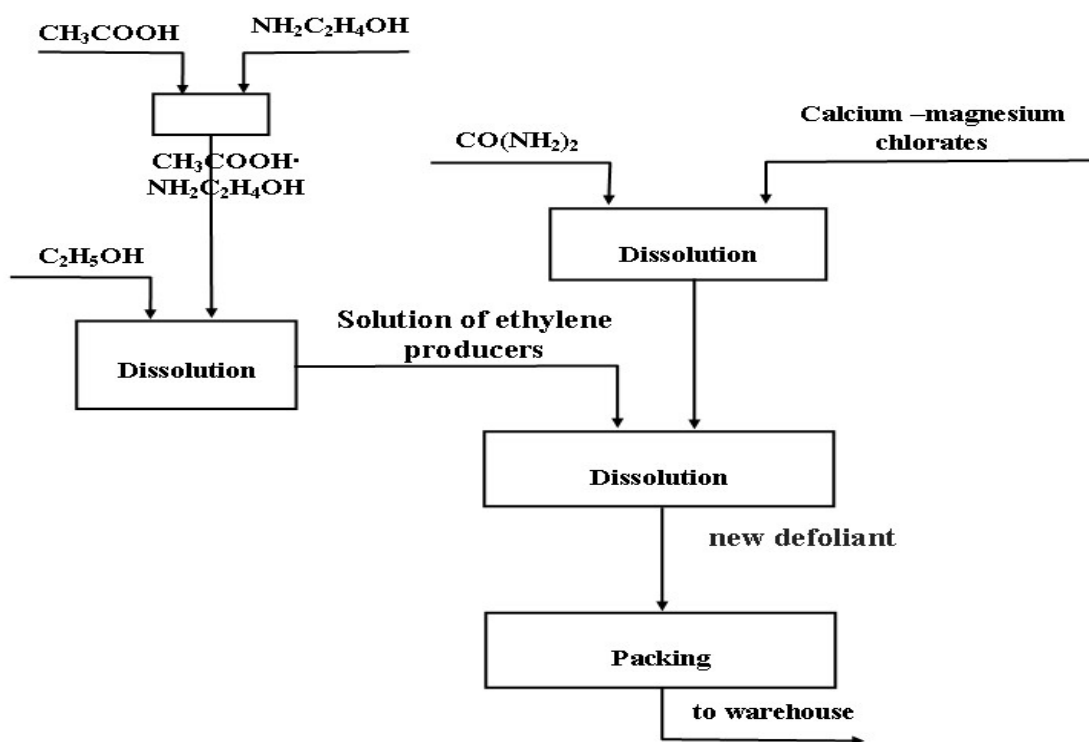


Fig. 2. Block diagram of obtaining 1 ton of defoliant of a new complex-acting defoliant

According to the technological diagram (Fig. 3), the calcium-magnesium chlorate preparation from the storage tank (1) is fed into the reactor by means of a pump (3) through a flow meter (4) (five). Here, through the hopper (6) and the belt weigher (7), the calculated amount of urea is fed. The dissolution of urea in a solution of calcium-magnesium chlorate preparation is carried out with constant stirring at a temperature of 30 ÷ 40 ° C. After dissolution of urea, a clear solution with a yellowish tint is formed, with a crystallization temperature of 1.9 ° C. The resulting solution flows by gravity into the intermediate tank (8). Then, by means of a pump (3), it is fed through a flow meter (9) to a reactor (10).

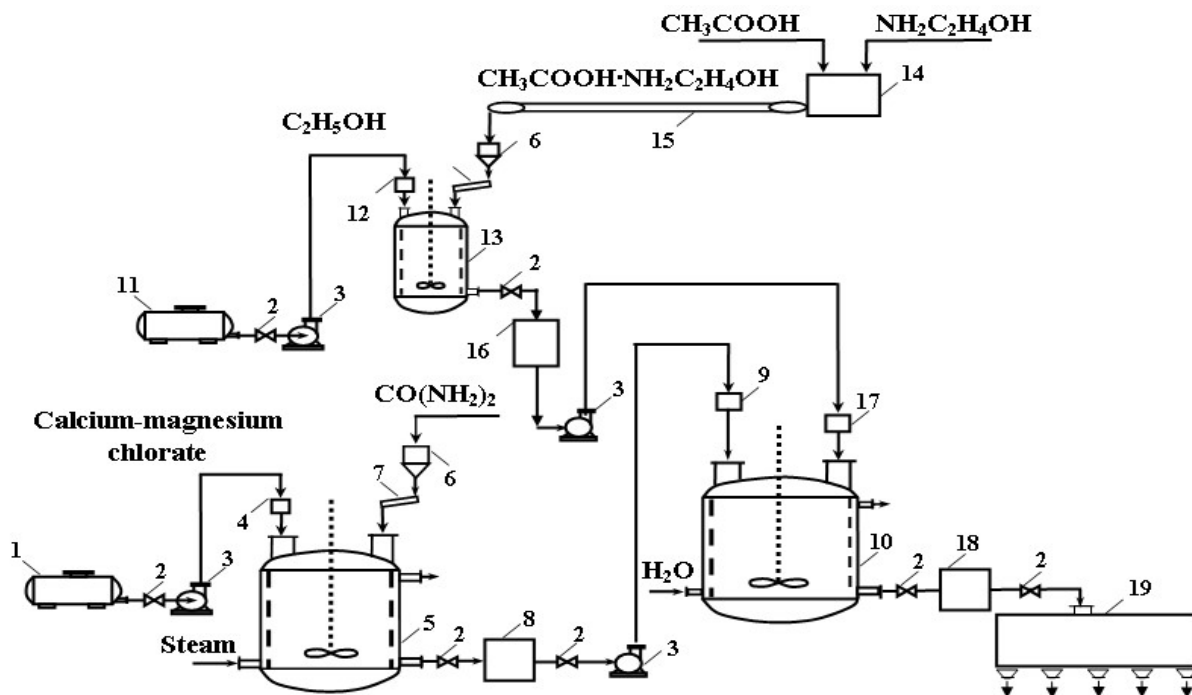


Fig. 3. Basic technological scheme for obtaining a new complex-acting defoliant

1,11-storage capacity; 2-valves; 3-centrifugal pumps; 4,9,12,17-flow meters; 6-bunker; 7-belt weighing batchers; 5,10,13-reactors; 15-belt conveyors; 8,16,18 intermediate tanks; 19-filling installation.

To obtain a solution of ethylene producers, ethanol from the storage tank (11) with the help of a centrifugal pump through a flow meter (12) enters the mixing reactor (13). The calculated amount of monoethanolamine acetate is fed there from the storage tank (16) through the flow meter (17). The process of dissolving ethyl acetate in ethanol is carried out by intensive stirring at a temperature of 20-25 ° C. The resulting solution flows by gravity into the intermediate tank (16) and through the flow meter (17) enters the reactor (10) with a mass ratio of a solution of calcium-magnesium chlorate preparation containing urea and a solution of ethylene producers of 1.0: 0.046. To avoid the evaporation of ethylene producers, the temperature in the reactor (10) is maintained within the range of 20 ÷ 25 ° C.

Table 2 : Physicochemical parameters of the preparation

Nº	Name of indicators	Norm
1	Appearance	Clear solution with a yellowish tinge
2	Mass fraction of the sum of calcium and magnesium chlorates,%	34,0÷35,0
3	Mass fraction of the sum of calcium and magnesium chlorides,%	5,0÷7,0
4	Mass fraction of urea,% $\{1\}$ Mass fraction of ethanol,%	8,0÷12,0
5	Mass fraction of monoethanolamine acetate,%	4,0÷8,0
6	Density, g / cm <sup>3</sup> , not less	0,2÷0,4
7	Name of indicators	1,45

In order to release a pilot batch of a new complex defoliant, the Technological Regulations and Organization Standard for the defoliant of the new complex defoliant Ts 04643516-15: 2017 have been developed.



The proposed technology has been tested on a large laboratory facility and on a pilot plant at Ferganaazot JSC with by removing the technological indicators for obtaining a new complex-acting defoliant and accumulated 1000 kg of pilot batches of defoliant.

Based on the results of testing the technology for obtaining a new complex-acting defoliant, Table 2 shows the norms of the technological regime, the material balance of obtaining the defoliant.

Table 3: Standards of the technological regime for obtaining a new complex-acting defoliant

Operation name	Duration, min.	Temperature, °C	Number of loaded reagents per 1 ton of product, kg.
loading a calcium-magnesium preparation into the reactor chlorate	15÷20	30÷40	856,0
loading carbamide into the reactor and dissolving it into calcium-magnesium chlorate preparations	10÷15	30÷40	100,0
loading ethanol into the reactor mixer	5÷10	20÷25	40,0
loading and dissolution of monoethanolamine acetate in ethanol	5÷10	20÷25	4,0
loading into the synthesis reactor of a solution of calcium-magnesium chlorate preparation containing urea and a solution of ethylene producers	20÷30	20÷25	<u>956,0+44,0</u> 1000
draining and packing the finished product	30÷40	20÷25	

### Conclusions

Thus, the developed conceptual technological scheme for obtaining a new composition of a complex defoliant using local raw materials will lead to economic benefits both in terms of the cost of drugs due to the localization of the raw material base, and in terms of defoliation efficiency, which consists in low consumption rates of the drug not a unit of sown area and the quality of defoliation.

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