

Production Of A New Cotton Defoliant

Ergashev Dilmurod Adiljonovich¹ , Khamdamova Shokhida Sherzodovna²

¹Doctor of Philosophy (PhD), docent of chemical technological department, Fergana Polytechnical Institute

²Doctor of Sciences (DSc), dosent of the chemical-technological department of the Ferghana Polytechnic Institute
Republic of Uzbekistan

Annotation: This article presents a technological scheme for obtaining a defoliant based on local raw materials. Chlorates and chlorides of calcium and magnesium were used as the main substances, and physiologically active substances were used to improve the quality of the defoliant. The economic benefit was established both in terms of cost and effectiveness of the defoliant.

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

Introduction. In the world, when growing crops, special attention is paid to the development of technology for the production and use of highly effective, low-toxic and harmless chemicals. Synthetic removal of cotton leaves using defoliants is essential for obtaining a high, high-quality yield and harvesting raw cotton in a short time. Successful defoliation with the removal of fiber leaves and improved fiber quality, resulting in the possibility of timely autumn activities. In this regard, it is of great importance to develop a technology for obtaining low-toxicity, positively affecting an increase in the yield of raw cotton and improving its quality, enriched with physiological active substances that do not harm the soil and the environment and have a complex effect.

The world receive 20 million tons of cotton fiber from plants occupying 30 million hectares of crops. About 200 million people in more than 80 countries around the world are involved in harvesting cotton on plantations. Another 60 million people are employed in various enterprises for the processing of cotton fiber into cotton cloth, as well as for the production of offal, seed oil or protein used in the production of feed for farm animals. The leading cotton producers today are China (4 million tons per year), the United States (about 4 million tons), India (2.5 million tons), Pakistan (1.5 million tons) and Uzbekistan (1.2 million tons). These five countries account for 65% of the world's total cotton production. The remaining 35% are produced in 70 countries of the world, of which Greece, Spain and Australia can be distinguished [1]. The defoliation of cotton is an integral element of the industrial technology of growing this crop, which allows solving the problems of harvesting, reducing product losses, and improving its quality.

At present, preparations for protecting agricultural crops, defoliants and plant growth regulators are mainly imported from abroad in the form of active ingredients and preparative forms. For the production of magnesium chlorate defoliant (contains 36% of the active substance) [2] at Ferganaazot JSC, the initial raw material source of bischofite (magnesium chloride) is imported from Volgograd (Russia) or Turkmenistan for foreign currency. This leads to an increase in the cost of the defoliant.

To solve this problem, the employees of the Institute of National Economy and Development of the Academy of Sciences of the Republic of Uzbekistan and FerPI have developed a technology for obtaining a new chlorate calcium-magnesium defoliant by using the products of hydrochloric acid decomposition of dolomites m. R. "Shorsu" and "Pachkamar" [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 [4-8].

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 [9]. As a result of the exchange reaction, a solution of calcium chlorate in acetone was obtained. After the separation of the acetone extract from the solid phase and the 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 according to the method described in [10].

When studying the system, the visual polythermal method [11] was used.

methods of analytical chemistry, in particular: chlorate ion was determined by volumetric permanganatometric method [2]; calcium and magnesium were determined by the volumetric complexometric method [12]. The content of elemental nitrogen, carbon, hydrogen was carried out according to the method [13].

Results and discussion. In order to substantiate the process of obtaining a new, more effective drug with high defoliation activity, "soft" action on cotton and accelerating the physiological processes of maturation of cotton crop on the basis of calcium-magnesium chlorate defoliant, carbamide, ethanol and ethyl acetate, the rheological properties of solutions in the system $\{[19.37\% \text{Ca}(\text{ClO}_3)_2 + 15.06\% \text{Mg}(\text{ClO}_3)_2 + 3.72\% \text{CaCl}_2 + 2.68\% \text{MgCl}_2 + 45.17\% \text{H}_2\text{O}] + 10.0\% \text{CO}(\text{NH}_2)_2 + 4.0\% \text{C}_2\text{H}_5\text{OH}\} - \text{C}_4\text{H}_8\text{O}_2$ [8]. To clarify the effect of the components on the physicochemical properties of solutions of the above system, the change in the crystallization temperature, pH of the medium, viscosity and density of solutions, depending on the

composition, was studied. On the basis of the data obtained on the study of the physicochemical properties of solutions, a "composition-properties" diagram of this system was constructed (Fig. 1, Table 1).

According to the data obtained, the "composition-crystallization temperature" diagram of the system is characterized by the presence of two branches crystallization with a clear break in the solubility curve (Fig. 1, curve 1). Crystallization $[84.3\% \Sigma \text{Ca}(\text{ClO}_3)_2 + \text{Mg}(\text{ClO}_3)_2 + 15.69\% \Sigma \text{CaCl}_2 + \text{MgCl}_2]$ continues to 0.45% ethyl acetate at -8.0°C . At this point, crystallization of $[84.3\% \Sigma \text{Ca}(\text{ClO}_3)_2 + \text{Mg}(\text{ClO}_3)_2 + 15.69\% \Sigma \text{CaCl}_2 + \text{MgCl}_2]$ and $\text{CO}(\text{NH}_2)_2$ occurs. With an increase in ethyl acetate concentration of more than 0.45%, $\text{CO}(\text{NH}_2)_2$ crystallizes in the system.

Analysis of the "composition-pH" and "composition-viscosity" diagrams (Fig. 1, curves 2, 3) shows that As ethyl acetate is added, the pH and viscosity of the solutions gradually increase. At the double point, the pH and viscosity of the solution are 4.55 and 9.04, respectively. Further, with an increase in the concentration of ethyl acetate more than 0.45%, i.e. in the urea crystallization region, the pH of the resulting solutions increases from 4.55 to 4.90, and the viscosity increases from 9.04 to 9.75 mm² / s.

Analysis of the "composition-density" diagram of the system (Fig. 1, curve 4) shows that as the concentration of ethyl acetate increases, the density of the newly formed solutions decreases from 1.4346 to 1.4140 g / cm³. Curve 4 of the composition-density diagram has a slight break. The crystallization branches of the sum of chlorates and chlorides of calcium and magnesium correspond to the density of solutions $1.4373 \div 1.4346 \text{ g / cm}^3$ (Table 1).

The values of the density of solutions $1.4346 \div 1.4140 \text{ g / cm}^3$ correspond to the crystallization branch of $\text{CO}(\text{NH}_2)_2$.

Fig. 1. "Composition-properties" diagram of the system $\{[19.37\% \text{Ca}(\text{ClO}_3)_2 + 15.06\% \text{Mg}(\text{ClO}_3)_2 + 3.72\% \text{CaCl}_2 + 2.68\% \text{MgCl}_2 + 45.17\% \text{H}_2\text{O}] + 10.0\% \text{CO}(\text{NH}_2)_2 + 4.0\% \text{C}_2\text{H}_5\text{OH}\} - \text{C}_4\text{H}_8\text{O}_2$

1-crystallization temperature, 2- pH, 3-viscosity, 4-density

To select the optimal ratio of components in the composition of the new defoliant, their agrochemical tests were carried out on cotton. The analysis of the obtained results showed that the composition obtained with the ratio $\{[19.37\% \text{Ca}(\text{ClO}_3)_2 + 15.06\% \text{Mg}(\text{ClO}_3)_2 + 3.72\% \text{CaCl}_2 + 2.68\% \text{MgCl}_2 + 45.17\% \text{H}_2\text{O}] + 10.0\% \text{CO}(\text{NH}_2)_2 + 4.0\% \text{C}_2\text{H}_5\text{OH}\} : \text{C}_4\text{H}_8\text{O}_2$ equal to 1.0: 0.002 ÷ 0.004. After the treatment of cotton with these formulations of the preparation on the 12th day, leaf fall was 89-90%, while in the variant (Zh.KhMD) this figure was 78.6%. In addition, the presence of ethyl acetate and ethanol in the composition of the new defoliant contributed to the acceleration of ripening and opening of cotton bolls. The increase in the opening of the capsules in comparison with ethanol was 9-12%.

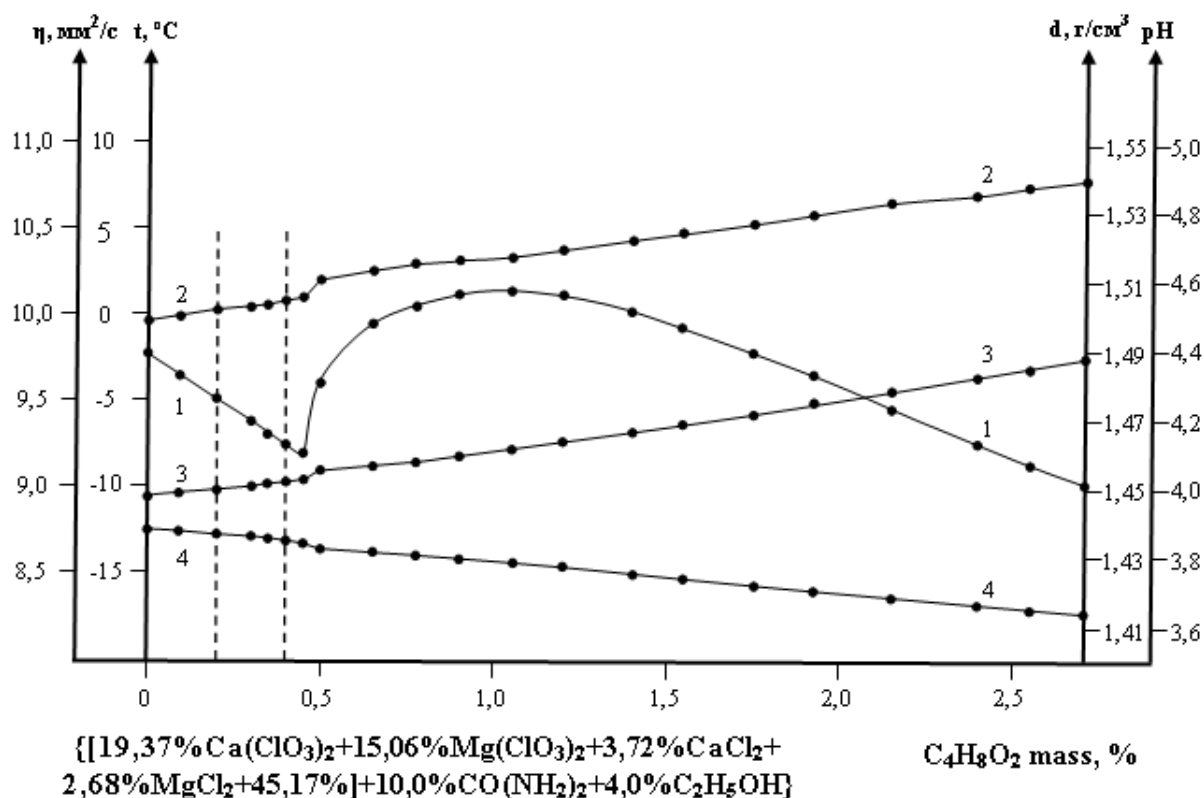


Fig. 1. "Composition-properties" diagram of the system $\{[19.37\% Ca (ClO_3)_2 + 15.06\% Mg (ClO_3)_2 + 3.72\% CaCl_2 + 2.68\% MgCl_2 + 45.17\% H_2O] + 10.0\% CO (NH_2)_2 + 4.0\% C_2H_5OH\} - C_4H_8O_2$
1-crystallization temperature, 2- pH, 3-viscosity, 4-density

Based on the results of studying the "composition-properties" of the above system and the conducted agrochemical tests of the defoliant compositions, it follows that to obtain an effective "mildly" acting drug with defoliation activity and accelerating the ripening of the crop is necessary in a solution of the preparation of the composition $\{[19.37\% Ca (ClO_3)_2 + 15.06\% Mg (ClO_3)_2 + 3.72\% CaCl_2 + 2.68\% MgCl_2 + 45.17\% H_2O] + 10.0\% CO (NH_2)_2 + 4.0\% C_2H_5OH\}$ dissolve ethyl acetate, at a mass ratio of 1.0: 0.002 ÷ 0.004. In this case, a defoliant solution with good physicochemical properties is formed, having a crystallization temperature - 4.8 ÷ -7.5 $^{\circ}C$, viscosity 8.98 ÷ 9.02 mm^2 / s , density 1.4365 ÷ 1.4348 g / cm^3 and pH 4.51 ÷ 4.54. The preparation contains: 34 ÷ 36% $\Sigma Ca (ClO_3)_2 + Mg (ClO_3)_2$; 10% $CO (NH_2)_2$; 4% C_2H_5OH ; 0.2 ÷ 0.4% $C_4H_8O_2$ the rest is water.

Table 1.

Dependence changes in crystallization temperature, pH, viscosity, density of solutions from the composition in the system

$\{[19.37\% Ca (ClO_3)_2 + 15.06\% Mg (ClO_3)_2 + 3.72\% CaCl_2 + 2.68\% MgCl_2 + 45.17\% H_2O] + 10.0\% CO (NH_2)_2 + 4.0\% C_2H_5OH\} - C_4H_8O_2$

№	Content of components, %		t, °C	pH	η, mm ² / c	d, g/sm ³
	{[19,37%Ca(ClO ₃) ₂ +15,06%Mg(ClO ₃) ₂ + 3,72%CaCl ₂ +2,68%MgCl ₂ +45,17%H ₂ O] +10,0%CO(NH ₂) ₂ +4,0%C ₂ H ₅ OH}	C ₄ H ₈ O ₂				
1	100	-	-2.3	4.47	8.94	1.4373
2	99.91	0.09	-3.6	4.49	8.96	1.4371
3	99.80	0.20	-4.8	4.51	8.98	1.4365
4	99.70	0.30	-6.3	4.52	9.00	1.4360
5	99.65	0.35	-7.0	4.53	9.01	1.4350
6	99.60	0.40	-7.5	4.54	9.02	1.4348
7	99.55	0.45	-8.0	4.55	9.04	1.4346
8	99.50	0.50	-4.0	4.60	9.10	1.4330
9	99.35	0.65	-0.5	4.63	9.12	1.4320
10	99.22	0.78	0.5	4.65	9.15	1.4308
11	99.10	0.90	1.2	4.66	9.18	1.4290
12	98.95	1.05	1.5	4.67	9.22	1.4280
13	98.80	1.20	1.0	4.69	9.27	1.4270
14	98.60	1.40	0	4.72	9.33	1.4250
15	98.46	1.54	-0.8	4.74	9.38	1.4240
16	98.25	1.75	-2.0	4.77	9.43	1.4220
17	98.08	1.92	-3.3	4.80	9.50	1.4208
18	97.85	2.15	-5.4	4.83	9.58	1.4180
19	97.60	2.40	-7.5	4.86	9.66	1.4165
20	97.45	2.55	-8.4	4.88	9.70	1.4150
21	97.29	2.71	-9.8	4.90	9.75	1.4140

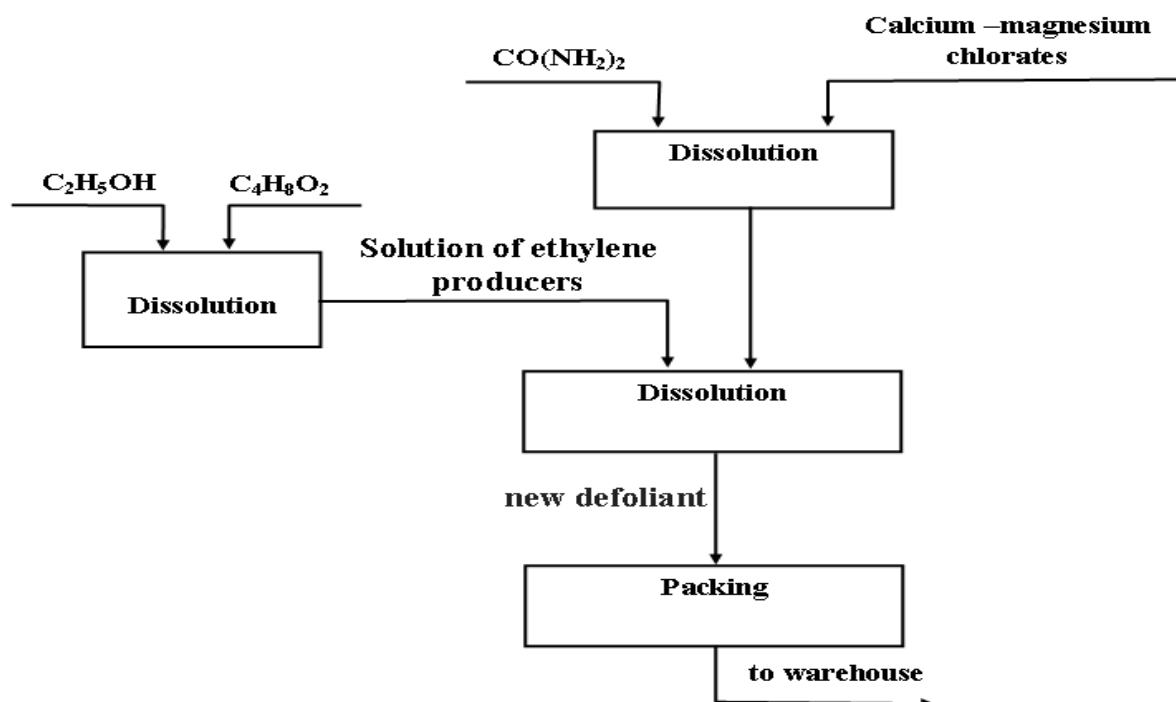
The production of a new defoliant, tentatively named "Fandef-alo", is carried out in a batch mode using crystalline carbamide, monoethanolamine acetate, as well as a solution of calcium-magnesium chlorate, ethanol and ethyl acetate. The essence of the method for the production of a new defoliant lies in the sequential dissolution of the calculated amounts of carbamide, ethanol and ethyl acetate (or monoethanolamine acetate) in a liquid calcium-magnesium chlorate preparation.

The production of the preparation tentatively named "Fandef-alo" consists of the following main stages: { {1} } - loading a solution of calcium-magnesium chlorate into the reactor;

- loading and dissolving carbamide in a solution of calcium-magnesium chlorate and obtaining a homogeneous solution;

- loading ethanol, ethyl acetate into the mixing reactor and dissolving the latter in ethanol;
- loading into the synthesis reactor a solution consisting of calcium-magnesium chlorate, carbamide and a solution of ethylene products, dissolving them to obtain a complex acting defoliant;
- packing the resulting product. {{ 1}}

Fig. 2. Block diagram for obtaining 1 ton of defoliant "Fandef-alo"



According to the technological scheme (Fig. 3), calcium-magnesium chlorate preparation from the storage tank (1) using a pump (3) through flow meter (4) enters the reactor (5). 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 is carried out with constant stirring at a temperature of $30 \div 40^\circ C$. After dissolution of urea, a clear solution with a yellowish tint is formed, with a crystallization temperature of $1.9^\circ C$. 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).

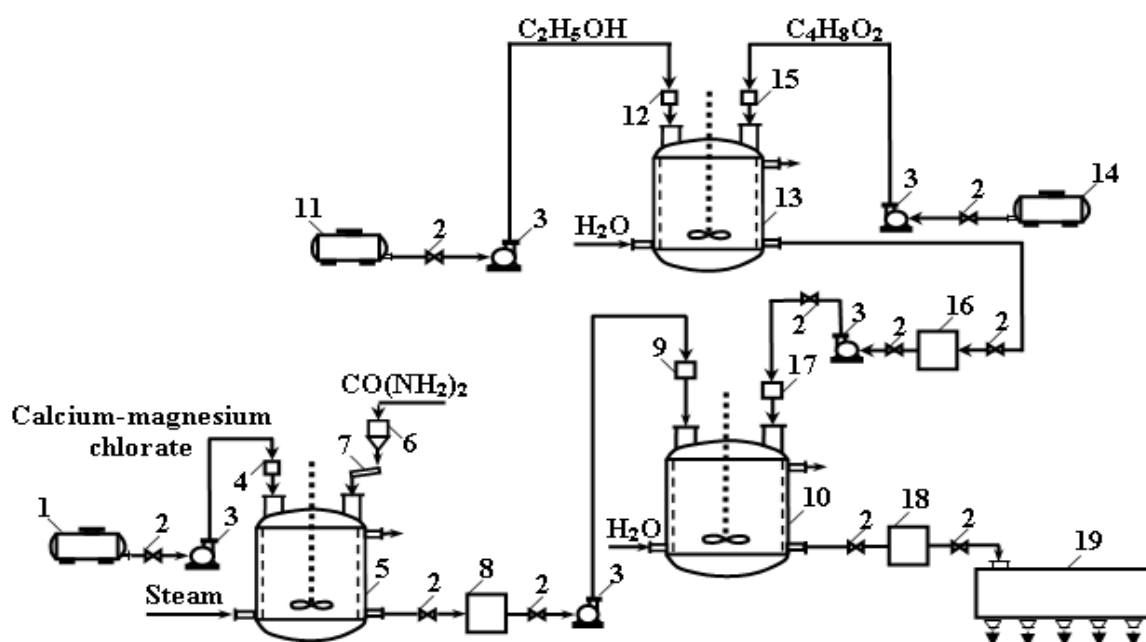


Fig. 3. Principal technological scheme for obtaining defoliant "Fandef-alo"

1,11,14-storage capacity; 2-valves; 3-centrifugal pumps; 4,9,12,15,17-flow meters; 6-bunker; 7-belt weighing batchers; 5,10,13-reactors; 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 ethyl acetate is supplied there from the storage tank (14) through the flow meter (15). 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). Then the resulting solution of ethylene producers from the intermediate tank (16) 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 1.0: 0.046. In order to avoid 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 drug "Fandef-alo"

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 ethyl acetate, %	4,0 ÷ 8,0

6	Density, g / cm ³ , not less	0,2÷0,4
7	Name of indicators	1,45

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

Table 3. Norms of technological mode for obtaining defoliant "Fandef-alo"

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 dissolving ethyl 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	

Ferghanaazot JSC with the removal of technological indicators for obtaining the defoliant "Fandef-alo" and 950 kg of pilot batches of defoliant were produced.

Based on the results of the development of the technology for obtaining the defoliant "Fandef-alo" defoliant.

Conclusions. Physicochemical foundations have been developed and a basic technological scheme for obtaining a new drug "Fandef-alo" has been proposed with its testing not by JSC "Ferganaazot", as well as the development of pilot batches of the drug. In the future, it is planned to conduct industrial agrochemical tests on various deciduous industrial crops. In case of obtaining high rates of defoliation and physiological

activity of the drug, it is planned to carry out the necessary measures to add the drug to the list of approved drugs by the State Chemistry Commission of the Republic of Uzbekistan [14].

References:

1. science-education.ru/ article/viewed=4578
2. Liquid chlorate magnesium defoliant. Technical conditions. Tsh 00203855-34: 2015. – 14 c.
3. Z.A. Khamrokulov, M.K. Askarova, S. Tukhtaev. Obtaining calcium-magnesium defoliant chlorate from dolomite //Chemical Journal of Kazakhstan. Almaty. 2015y №3 C. 164-170.
4. D.A.Ergashev, Sh.Sh.Khamdamova, A.N.Mirzaolimov, S.B.Mukhammedov. Obtaining calcium and magnesium chlorides from the dolomite of the Navbakhor deposit // Universum: Technical Sciences: electron. scientific. journal. November, 2019. № 11 (68). C. 68-74.
5. D.A.Ergashev, A.S.Togasharov, S.Tukhtaev, M.K.Askarova. Solubility of components in the system $[21,8\%Ca(ClO_3)_2+19,5\%Mg(ClO_3)_2+3,7\%CaCl_2+ 3,7\%MgCl_2+51,3\%H_2O]-CO(NH_2)_2-H_2O$ //Uzbek Chemical Journal№5 2012 r. C. 34-39.
6. D.A.Ergashev. Study of rheological properties of solutions in the system $\{[20,26\%Ca(ClO_3)_2+15,76\%Mg(ClO_3)_2+3,9\%CaCl_2+2,81\%MgCl_2+47,27\%H_2O] + 10\%CO(NH_2)_2\} - C_2H_5OH$ //Universum: Technical sciences: electron. scientific. journal. 2016. № 6 (27).
7. R.Nazirov, S.Tajiev, S.Mirsalimova, Sh.Hamdamaova. //Intensification of the process of obtaining complex fertilizers from local raw materials//.Monograph. ed. by B.S.Zakirov. - Ufa: Omega science, 2019, 126 c. <https://www.elibrary.ru/item.asp?id=41588683>
8. 8. M.Sabirov, R.Nazirov, Sh.Hamdamaova, S.Tajiev.//Intensification of the process of obtaining complex suspended fertilizers with insecticidal activity//. Monograph. Ferghana-Vinnytsia: NGO "European Scientific Platform", 2020. 137 c. <https://doi.org/10.36074/tad-sob-naz-ham.monograph>
- 9.Ikramov, M., Nazirov, R., Mirsalimova, S., Tajiev, S. //New types of suspended fertilizers based on local raw materials//. Monograph. Ferghana-Vinnytsia: NGO "European Scientific Platform», 2020. 123 c. <https://doi.org/10.36074/ik-na-mi-ta.monograph>
10. D.A.Ergashev, M.K.Askarova, S.Tukhtaev. Studying the mutual influence of components in the system $[22,52\%Ca(ClO_3)_2+17,51\%Mg(ClO_3)_2+4,33\%CaCl_2+ 3,12\%MgCl_2+52,52\%H_2O] - C_2H_5OH - H_2O$ // "Chemical technology. Control and Management" International Scientific and Technical Journal №4 2016r. C. 10-13.
- 11.11. D.A.Ergashev, M.K.Askarova, S.Tukhtaev. Dependence of changes in the physico-chemical properties of solutions on the composition in the system $\{[19,37\%Ca(ClO_3)_2+15,06\%Mg(ClO_3)_2+3,72\%CaCl_2+2,68\%MgCl_2+45,17\%H_2O]+10,0\%CO(NH_2)_2+$

12.4,0% C_2H_5OH } – $C_4H_8O_2$ /"Chemical technology. Control and Management" International Scientific and Technical Journal№2 2017r. C. 50-53.

13.A.c. 1143691 the USSR. Method of obtaining chlorate-chloride of calcium defoliant / M.N. Nabiev, R. Shammassov, S. Tukhtaev, H. Kucherov, etc. (USSR)-No. 3620951/23-26; announced 23.05.83.; publ. 07.03.85 // Discoveries, inventions.- 1985. -No. 9. -p.84.

14.MartynovYu.M., Matveev M.A., Yakimenko L.M., Furman A.A. Technology of production and application of chlorate-magnesium defoliants. // Chemical industry. 1958. No. 7. With. 420-423.

15.15. Trunin A.S., Petrova D.G. Visual - polythermal method. Kuibyshev Polytechnic University. In-t /:- Kuibyshev, 1977. -94c./ Dep. in VINITINº584-78.

16.16. Schwarzenbach G., Flashka G. Complexometric titration. - M.: Chemistry, 1970. -360c.

17.Klimova V.A. Basic micromethods for the analysis of organic compounds. M.: Chemistry, 1975. - 224c.

18.List of chemical and biological means of pest control, plant diseases and weeds, defoliants and plant growth regulators approved for use in agriculture of the Republic of Uzbekistan, Tashkent-2016 p.306-309.

19. Adilov, Z., Ergashev, D., Tozhiev, R., &Khamdamova, S. Preparation of chlorate-containing defoliants with insecticidal properties. Monograph. Ferghana-Vinnytsia: NGO "EuropeanScientificPlatform",2021. 123 c. . <https://doi.org/10.36074/ad-er-to-kha.monograph>