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= 1.940=4.260= 0.350

QR - Issue

QR - Article



p-ISSN: 2308-4944 (print) e-ISSN: 2409-0085 (online)

Year: 2021 Volume: 102 Issue: 10

http://T-Science.org Published: 29.10.2021





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DEVELOPMENT OF LITTLE TOXIC LITTLE-TOXIC DEFOLIANTS **BASED ON 2-CHLOROETHYL PHOSPHONIC ACID -**AMINOGUANIDINE CARBONATE - WATER

Abstract: The solubility of the $ClCH_2CH_2PO(OH)_2 - NH_2NHCNHNH_2 * H_2CO_3 - H_2O$ system has been studied in the temperature range from freezing -47.6 °C to 40 °C. A polythermal solubility diagram has been constructed, on which ice crystallization fields, ClCH₂CH₂PO(OH)₂, NH₂NHCNHNH₂ * H₂CO₃, and compounds of the composition CICH₂CH₂PO(OH)₂·NH₂NHCNHNH₂. are distinguished. The compound was isolated from the putative crystallization region and identified by chemical and physicochemical analysis methods.

Key words: solubility diagram, 2-chloroethyl phosphonic acid, aminoguanidine carbonate, effective defoliants, concentration, system, polytherm.

Language: English

Citation: Shakarov, N. J., Ergashev, I. Sh., & Nomirov, M.N. (2021). Development of little toxic little-toxic defoliants based on 2-chloroethyl phosphonic acid - aminoguanidine carbonate - water. ISJ Theoretical & Applied Science, 10 (102), 969-974.

Soi: http://s-o-i.org/1.1/TAS-10-102-110 Doi: crossee https://dx.doi.org/10.15863/TAS.2021.10.102.110

Scopus ASCC: 1600.

Introduction

UDC 547.495.9

Cotton-growing in the country is one of the most important branches of agriculture. Today, the primary task of technical progress in cotton-growing is the mechanized harvesting of uranium, in the implementation of which an extremely important role plays the pre-harvest chemical defoliation of cotton with the help of chemical preparations (defoliants). Without this important agrotechnical event, it is

impossible to achieve the high productivity of cotton harvesting machines and the success of cotton growing at the present stage of cotton cultivation.

To successfully solve the problem of artificial leaf removal, it is necessary to have highly effective defoliants, which ensure cotton leaf leaves more than 80-85% of a single treatment at a low consumption rate, which act "softly" on plants, and therefore do not negatively affect them and not decrease the harvest, it's quality and oil content of seeds, as well as nonclogging cotton fibre.



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Materials and methods

One of the most valid indicators of defoliants, causing wide practical application of preparations, is their environmental safety from the point of view of environmental protection.

Thus, the data on the solubility of the components in the system 2-chloroethyl phosphonic acid – aminoguanidine carbonated – water does not exist. However, the results of the interaction of these substances in the aquatic environment allow not only to reveal the chemistry of the reacting components, but also contribute to their practical use for the preparation of defoliants based on these compounds and their complexes.

Therefore, to characterize the behaviour of the initial components in polythermal conditions, this system was studied from - 47.6 to 40 °C. The binary system of aminoguanidine carbonated - water that make up this system, we studied from - 0.1 to 70 °C. The solubility diagram of it consists of two branches of crystallization of the initial components intersecting at a eutectic point, which is 0.1% aminoguanidine carbonate at - 0.1 °C.

The $ClCH_2CH_2PO(OH)_2 - NH_2NHCNHNH_2 * H_2CO_3 + H_2O$ system was studied using ten internal cuts from full freezing temperature (-47.6 °C) to 40 °C (Fig. 1).

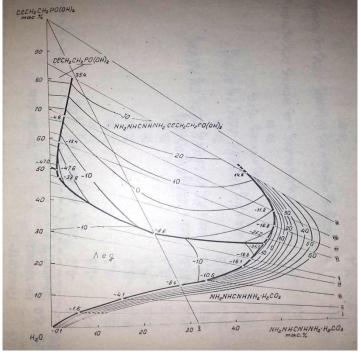


Figure.1. System solubility diagram ClCH₂CH₂PO(OH)₂- NH₂NHCNHNH₂* H₂CO₃ + H₂O

On the polythermal diagram, the solubility delimits the crystallization fields of ice, aminoguanidine carbonate, 2-chloroethyl phosphonic acid and NH₂NHCNHNH₂ * ClCH₂CH₂PO(OH)₂ compounds for which the temperature and

concentration limits of existence were determined. The fields converge at two nodal points corresponding to the crystallization of three different solid phases (Table 1).

Table 1. Double and triple points of the system 2 - chloroethyl phosphonic acid- aminoguanidine carbonate – water

The liquid phase composition, wt.%			Crystallization	Solid phases	
NH ₂ NHCNHNH ₂ *H ₂ CO ₃	ClCH ₂ CH ₂ PO(OH) ₂	H_2O	temperature, °C		
0,1	-	99,9	-0,1	Ice+ NH2NHCNHNH2*H2CO3	
4,7	6,0	89,3	-1,6	The same	
8,5	15,4	76,1	-4,1	The same	
17,3	42,4	40,3	-16,1	The same	
19,6	44,2	36,2	-18,8	The same	
12,0	28,1	59,9	-8,4	The same	



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-	50,4	49,6	-47,0	$Ice + ClCH_2CH_2PO(OH)_2$	
28,3	22,9	48,8	0.1	Ice + NH ₂ NHCNHNH ₂ *	
26,3		40,0	-8,4	ClCH ₂ CH ₂ PO(OH) ₂	
47,1	1,9	51,0	-38,8	То же	
				Ice + NH ₂ NHCNHNH ₂ *	
24,4	47,5	28,1	-25,8	ClCH ₂ CH ₂ PO(OH) ₂ +	
				NH ₂ NHCNHNH ₂ *H ₂ CO ₃	
25,1	47,8	27,1	-25,2	The same	
			-47,8	Ice + ClCH ₂ CH ₂ PO(OH) ₂ +	
1,6	49,6	48,8		NH ₂ NHCNHNH ₂ *	
				ClCH ₂ CH ₂ PO(OH) ₂	
				ClCH ₂ CH ₂ PO(OH) ₂ +	
67,5	3,6	28,9	3,6	NH ₂ NHCNHNH ₂ *	
				ClCH ₂ CH ₂ PO(OH) ₂	
80,4	5,6	14,0	35,4	The same	
58,8	2,2	39,0	-15,4	The same	
				NH ₂ NHCNHNH ₂ *	
29,9	50,0	20,1	-18,8	ClCH ₂ CH ₂ PO(OH) ₂ +	
				NH ₂ NHCNHNH ₂ *H ₂ CO ₃	
47,0	44,8	8,2	14,8	The same	
34,8	50,1	15,1	-11,2	The same	

On the polythermal diagram, the solubility isotherms are plotted every 10 °C. To clarify the coordinates of the nodal points of the system, its projections onto the corresponding water sides of the concentration triangle were constructed.

According to the solubility diagram of the studied system, the formation of the compound NH₂NHCNHNH₂* ClCH₂CH₂PO(OH)₂ in the system occurs in the temperature range - $47.6 \div 35.4$ °C. The minimum concentration of aminoguanidine carbonate causing the formation of aminoguanidine 2-chloroethyl phosphonic in the system, 1.6% NH₂NHCNHNH₂* ClCH₂CH₂PO(OH)₂ is a white crystalline substance with a melting point of 140 °C. The solubility of aminoguanidine 2-chloroethyl phosphonate in water was studied, it was established

that it is highly soluble in water and its solubility at 0, 10, 20 °C is 53.6 - 65.2 - 72.1%, respectively.

The compound was identified by chemical, X-ray phase, derivatographic and IR - spectroscopic methods of analysis.

The results of chemical analysis: found, mol,%: 66.05 - ClCH₂CH₂PO(OH)₂, 33.95 - NH₂NHCNHNH₂. For NH₂NHCNHNH₂* ClCH₂CH₂PO(OH)₂, mol%, 66.13 - ClCH₂CH₂PO(OH)₂, 33.87 - NH₂NHCNHNH₂ was calculated.

X-ray phase analysis confirms the formation of the indicated compounds in the system, which is distinguished by its own diffraction reflexes, which are not characteristic for the initial components (Fig. 2.).

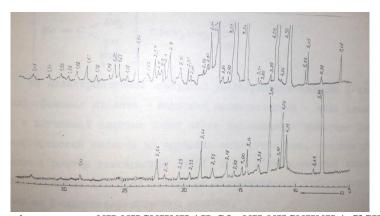


Figure 2. Diffraction patterns: NH₂NHCNHNH₂*H₂CO₃ NH₂NHCNHNH₂* ClCH₂CH₂PO(OH)₂

The compound NH₂NHCNHNH₂* CICH₂CH₂PO(OH)₂ is characterized by endothermic

decomposition, as evidenced by the data of thermal analysis (Fig. 3).



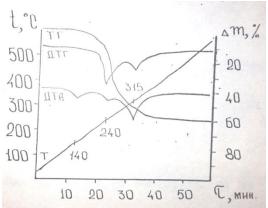


Figure 3. NH2NHCNHNH2*ClCH2CH2PO(OH)2 derivatogram

When heated in atmospheric air, the compound melts congruently at $140~^{\circ}\text{C}$ with the formation of a homogeneous melt. At $240~^{\circ}\text{C}$ it decomposes with the removal of 45.33% of the substance. The endothermic effect at $315~^{\circ}\text{C}$ corresponds to further decomposition of the compound. On the TG derivatograms, the mass loss, in this case, is 14.67%.

In the infrared spectrum of aminoguanidine 2-chloroethyl phosphonate, a significant shift of absorption band of the valence antisymmetric and symmetric NH₂ - group oscillations occurs and is observed in the frequency range 3015–2930 cm⁻¹, which indicates their participation in the formation of new bonds with 2-chloroethyl phosphonic acid.

The absorption bands characteristic for $\alpha(C=N)$ are appeared in the frequency range of 1600-1565 cm⁻¹ and practically do not change their position in comparison with the IR spectrum of aminoguanidine. Given these circumstances, the structure of NH₂NHCNHNH₂*ClCH₂CH₂PO(OH)₂ can be assumed as follows:

From the polythermal solubility diagram of the system $\,2\,$ - chloroethyl phosphonic acid -

aminoguanidine carbonate - water, it follows that in the studied system there is a salting-out effect of 2chloroethyl phosphonic acid on aminoguanidine carbonate, which decreases with increasing temperature, while aminoguanidine carbonate, on the contrary, has a slight salting-out effect on 2chloroethyl phosphonic acid.

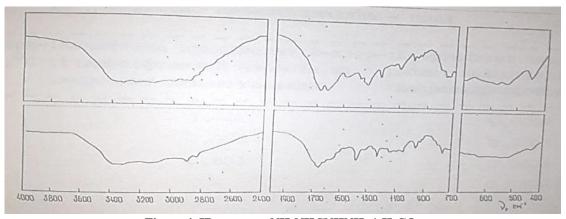


Figure 4. IR spectra - NH₂NHCNHNH₂ * H₂CO₃ NH₂NHCNHNH₂ * ClCH₂CH₂PO(OH)₂



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$\mathbf{JIF} = 1.500$	SJIF (Morocco) = 7.184	OAJI (USA)	= 0.350

Thus, in the presented chapter, the mutual solubility of the components in four aqueous systems based on 2-chloroethyl phosphonic acid and aminoguanidine salts with inorganic acids is considered. They are of a simple eutonic type, with the exception of the systems: CICH₂CH₂PO(OH)₂ - NH₂NHCNHNH₂* H₂CO₃ - H₂O, CICH₂CH₂PO(OH)₂ - 2NH₂NHCNHNH₂* H₂SO₄ * H₂O - H₂O in which, as a result of interaction between

the components occurs the formation of the compounds with the composition $NH_2NHCNHNH_2*$ $CICH_2CH_2PO(OH)_2$ and $NH_2NHCNHNH_2*$ H_2SO_4* H_2O_2

For these compounds, the temperature and concentration limits of formation in the systems have been established, their identification was given (Tab.2).

Table 2. Salting out coefficient and salting constant of the components in the system 2 - chloroethyl phosphonic acid - aminoguanidine carbonate – water.

Temperature, °C	Salting constant of 2 - chloroethyl phosphonic acid on aminoguanidine carbonate	Salting coefficient of aminoguanidine carbonate on 2 - chloroethyl phosphonic acid
0	1,267	0,0002
10	1,079	0,0003
20	0,801	0,0004
30	-	0,0009

In all systems, aminoguanidine salts have a salting-out effect on 2-chloroethyl phosphonic acid, which increases in the order NH₂NHCNHNH₂* H₂CO₃ \langle NH₂NHCNHNH₂* HNO₃ \langle 2NH₂NHCNHNH₂ * H₂SO₄ * H₂O \langle NH₂NHCNHNH₂ * H₃PO₄.

While 2 - chloroethyl phosphonic acid, on the contrary, has a salting-out effect on aminoguanidine salts, in addition to NH₂NHCNHNH₂* H₂CO₃.

The salting-out effect on aminoguanidine salts increases in the order NH₂NHCNHNH₂* HNO₃ < 2NH₂NHCNHNH₂ * H₂SO₄ * H₂O < NH₂NHCNHNH₂* H₂CO₃.

Conclusion

Analyzing the study results of the interaction of 2-chloroethyl phosphonic acid with the salts of guanidine and aminoguanidine in the corresponding systems, it should be noted that in systems with carbonate and sulphate salts, as a rule, chemical interaction occurs between the components with the formation of compounds of identical composition NH₂NHCNHNH₂ * ClCH₂CH₂PO(OH)₂, NH₂NHCNHNH₂ * H₂SO₄, NH₂NHCNHNH₂ * H₂SO₄* H₂O. In systems with the participation of guanidine salts, mutual salting out of the components at each other occurs, and with the inclusion of aminoguanidine salts, their salting-out effect on 2-chloroethyl phosphonic acid is observed.

In the presence of 2-chloroethyl phosphonic acid, all aminoguanidine salts, with the exception of $NH_2NHCNHNH_2 * H_3PO_4$ increase their solubility.

The salting-out effect on 2 - chloroethyl phosphonic acid of the studied salts decreases upon transition from the salts of guanidine to aminoguanidine.

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