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INVESTIGATION OF THE SYNTHESIS OF ACETYLENE AMINO ALCOHOLS AND THE STUDY OF THEIR BIOLOGICAL ACTIVITY

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Abstract: This work describes the synthesis of acetylene amino alcohols based on the aminomethylation of secondary acetylene alcohols in a 1,4-dioxane solvent medium, using formaldehyde and hydroxyl-containing or heterocyclic amino compounds under the catalysis of copper(I) salts. Hex-1-yn-3-ol was used as a secondary acetylene alcohol, and amines such as monoethanolamine, diethanolamine, and morpholine were used as aminating agents. As a result of the conducted research, amino alcohols with high yields were synthesized. The results of studies on the use of the obtained acetylene amino alcohols in agriculture are presented. The biostimulating effect of synthesized acetylene amino alcohols has been studied. Their positive effect on the germination of wheat seeds and an increase in yield has been established.

Keywords: secondary acetylene alcohol, hex-1-yn-3-ol, acetylene amino alcohol, Mannich reaction, formaldehyde, hydroxyl-containing and heterocyclic amines, biostimulator, seed germination, wheat growth.

Introduction. Many nitrogen-containing compounds synthesized on the basis of secondary acetylenic alcohols exhibit physiological activity and, due to this, are used in various fields of industry and agriculture as herbicides, fungicides, defoliants, growth regulators, pharmaceuticals, corrosion inhibitors, and more. The biological activity of these compounds is explained by the presence of several reactive sites in their molecules (acetylene bond, hydroxyl group, heterocyclic ring, and amino group) [1–5].

In several studies, the most convenient and widespread method of obtaining acetylene amino alcohols involved the Mannich reaction, in which acetylene alcohols were aminomethylated using formaldehyde and a secondary amine. Hydroxyl-containing and heterocyclic amines such as ethanolamine, diethanolamine, and morpholine were selected as aminating agents.

Materials and Research Methods: Through the condensation of n-oil-aldehyde or propanone [4,5] with acetylene, the corresponding acetylenic alcohols were obtained. The influence of various factors (temperature, pressure, reaction duration, and the nature of the solvent used) on the synthesis process was investigated. The resulting compounds were then subjected to the Mannich reaction, where they were converted into the corresponding amino alcohols by aminomethylation using mixtures of paraformaldehyde with monoethanolamine, diethanolamine, and morpholine, as shown in the following scheme:

$$\underset{\displaystyle\mathrm{OH}}{\mathrm{R{-}\underset{|}{C}H{-}C{\equiv}CH}} + CH_2O + NH(R')_2 \xrightarrow[\text{dioxan}]{Cu^+,\ ^{o}t} \underset{\displaystyle\mathrm{OH}}{\mathrm{R{-}\underset{|}{C}H{-}C{\equiv}C{-}CH_2{-}N(R')_2}} + H_2O$$

where: -N(R′)$_2$ = monoethanolamine, diethanolamine, or morpholine group

The synthesis of acetylene amino alcohols was carried out according to the scheme at 90–100 °C, catalyzed by copper(I) acetate salts in a 1,4-dioxane solvent medium [5–8].

0.1 mol of acetylene alcohol, 0.1 mol of paraformaldehyde, 80 ml of dioxane, and 0.02 g of copper(I) chloride were placed in a 250 ml four-necked flask equipped with a stirrer, condenser, and thermometer. The reaction mixture was stirred intensively at 90–100 °C for 6–7 hours.

Within 30 minutes of starting the reaction, a mixture of 50 ml of 1,4-dioxane and 0.1 mol of secondary amine was added through a funnel. After the reaction was complete, the mixture was left overnight. The following day, the contents were transferred to a 1000 ml separation funnel, diluted with distilled water, and extracted with 200 ml of chloroform. The organic layer (main product) was isolated. This process was repeated at least three times.

The chloroform layers were combined and dried with potassium carbonate. Chloroform was removed by distillation in a water bath at 60–80 °C. The residue was separated into components by vacuum distillation.

Acetylene alcohols, amino alcohols, and hydroxy acids act as biostimulants for plants [5–8]. The acetylene amino alcohols synthesized were tested for their ability to stimulate plant seed germination, control plant growth, and increase yield. Treatments included soaking seeds in aqueous solutions of the compounds.

In the next stage, treatments involved spraying plant stems with the same solutions. These tests were conducted jointly with the Namangan Scientific Experimental Station for grain and leguminous crops in wheat-planted experimental fields.

The biostimulating properties of synthesized acetylene amino alcohols – 1-((2-hydroxyethyl)amino)hept-2-in-4-ol (GEAG), 2,2'-((4-hydroxyhept-2-in-1-yl)azanediyl)bis(ethane-1-ol) (GGAE), and 1-morpholinohept-2-in-4-ol (MG) – were studied on Babur variety wheat crops sown in autumn on 1.2 hectares of experimental plots.

Germination rate depends on climatic and soil conditions, physical and chemical soil properties, the effect of stimulants, fertilizer type and dosage, seed variety, and sowing time [1–5].

200 kg of grain seeds were used per hectare, with 0.2 kg used per 40 m^2 plot. Seeds in five different variants were soaked for one day in 0.05% aqueous solutions of GEAG, GGAE, MG, and succinic acid (as reference).

Under laboratory conditions, 0.05% concentrations of these compounds were optimal. Therefore, 0.005% solutions were used in field trials. Wheat seeds were soaked in 0.05% aqueous solutions for four days before sowing.

Results and Discussion: The experimental results showed that favorable synthesis conditions yielded high amounts of acetylene amino alcohols using hydroxyl-containing and heterocyclic amines. Maximum yield was obtained with a 6-hour reaction at 95 °C. The yield from heterocyclic amines was higher than from hydroxyl-containing ones. Yields and physicochemical constants are shown in Table 1.

Table 1. Yields and physicochemical constants of synthesized acetylenic amino alcohols

№	Acetylenic amino alcohol	Yield (%)	Boiling Point (°C/mmHg) or Melting Point (°C)	Density (g/cm³)	Refractive Index (n ± 0.003)
I	GEAG	54.4	140–141 / 76	0.9720	1.4783
II	GGAE	62.7	158–160 / 76	0.9910	1.4800
III	MG	70.2	148–150 / 76	0.9082	1.4721

During field experiments, the number of sprouted seedlings was counted 12 days after sowing. The results are shown in Table 2.

Table 2. Effect of preparations on wheat seed germination

№	Treatment Variant	Germination (%)	Difference from Control (%)
1	Control (water)	87.3	–
2	0.05% GEAG solution	93.6	+6.3
3	0.05% GGAE solution	94.2	+6.9
4	0.05% MG solution	94.1	+6.8
5	0.05% succinic acid solution	89.7	+2.4

Table 3. Effect of preparations on wheat growth and yield

№	Treatment Variant	Yield (c/ha)	Increase over Control (c/ha)
1	Control	55.8	–
2	0.05% GEAG solution	60.8	+5.0
3	0.05% GGAE solution	61.8	+6.0
4	0.05% MG solution	61.6	+5.8
5	0.05% succinic acid solution	58.7	+2.9

The results confirm that the new preparations positively affected the yield of Babur variety winter wheat, increasing yield by 5.0–6.0 c/ha over the control.

Conclusion. Based on the study, compounds containing unsaturated bonds and amino and hydroxyl groups demonstrate high biological activity. Acetylene amino alcohols with such reactive centers act as effective biostimulants, promoting plant growth and development. They positively affect seed germination and seedling growth, ultimately improving yield. Moreover, these compounds enhance plant resistance to environmental stress and diseases.

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