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# STUDY OF THE CATALYTIC SYNTHESIS OF O-VINYL ETHER BASED ON MONOETHANOLAMINE AND ACETYLENE

**JURABOEV FOZIL**

PhD of Namangan institute of engineering and technology
E-mail: jfm-75@mail.ru

**TURSUNOV BAKHTIYOR**

Master student of the Namangan Engineering and Technology Institute

**TOGAEVA MAFTUNA**

Student of the Namangan Engineering and Technology Institute

**Abstract:**

**Objective.** Synthesis of vinyl esters by vinylation of monoethanolamine with acetylene in superbasic medium was investigated. The effect of temperature, catalyst, reaction time, and other factors on the yield of vinyl ether of monoethanolamine has been studied. Also, quantum-chemical calculations theoretically calculated the active reaction centers of monoethanolamine, the distribution of atomic charges.

**Methods.** Based on literature analysis, monoethanolamine was exposed to acetylene with KOH catalyst, DMSO solvent medium, acetylene pressure 4 atm, temperature 80-95°C for 5-7 hours. The spatial structure of the monoethanolamine molecule and the distribution of atomic charges were studied using the Chem 3D Ultra 10.0 program.

**Results**. The yield of vinyl ester of monoethanolamine is significantly affected by the reaction temperature and time. The yield of O-vinyl ether increased with increasing temperature from 80°C to 90°C and increasing the reaction time from 3 to 6 hours. When the temperature rises to 95°C and the vinylization process is extended to 7 hours, the product yield decreases. This situation is explained by a decrease in the solubility of gaseous acetylene in the solvent and by the processes of oligomerization of various vinyl esters.

**Conclusions.** It was found that monoethanolamine and acetylene give o-vinyl ether with a maximum content of 54.6% for 6 hours at a temperature of 90 ° C and a pressure of 4 atm.

**Keywords:** monoethanolamine, vinylation process, vinyl ether, hydroxyl group, nucleophilicity, charge distribution, reaction time, yield of monoethanolamine vinyl ether.

**Introduction.** Today, topical issues are the synthesis of new types of organic substances, obtaining compounds with different properties on their basis by introducing modern technologies in the chemical industry. In this regard, important issues are the creation of various biologically active chemicals, pharmaceuticals with unique properties, polymeric substances, adhesives and paints, which are widely used in the cultivation of agricultural products. These compounds include simple and complex vinyl ethers containing hydroxyl, carboxyl and amino groups in the molecule, vinylized with acetylene [1-8].

**Methods.** Vinyl compounds synthesized in the world in recent years are widely used in agriculture and pharmaceuticals due to their high physiological activity. Vinyl ethers have been synthesized in various ways. In particular, the synthesis of vinyl ethers and esters by vinylation of compounds containing hydroxyl and carboxyl groups – alcohols, carboxylic acids, and hydroxy acids—with acetylene has been well studied. In particular, the synthesis of vinyl ethers in an alkaline medium according to the Favorsky-Shostakovsky method is widely used [9-14].

Obtaining vinyl derivatives by transferring a hydrogen atom of the hydroxyl group to the triple bond of acetylene is one of the most common methods in organic synthesis. The use of

superbasic media in this method serves as an important factor in improving the performance of the product [9-16].

Although the vinylation of alcohols, acids, and hydroxy acids with acetylene has been relatively well studied, the processes of vinylation of amino alcohols have been less studied.

The presence of amino groups and hydroxyl groups in the monoethanolamine molecule complicates the processes of vinylation. Because in vinylation processes, the vinyl group can be replaced by an amino group hydrogen or a hydroxyl group hydrogen. Therefore, the molecular structure and charge distribution were theoretically calculated using Chem 3D Ultra 10.0 software to predict the course of vinylation reactions.

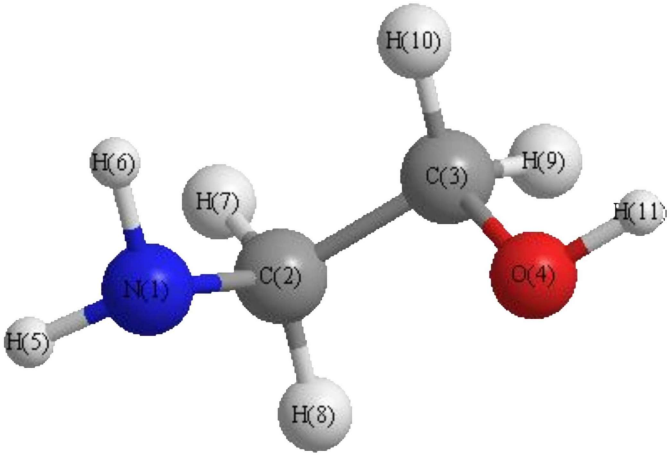


**Figure 1. Spatial structure and charge distribution of the monoethanolamine molecule**

*Here,* -0.284 [N(1)]; 0.031 [C(2)]; 0.151 [C(3)]; **-0.374 [O(4)]**; 0.110 [H(5)]; 0.110 [H(6)]; 0.009 [H(7)]; 0.024 [H(8)]; 0.012 [H(9)]; 0.012 [H(10)]; 0.199 [H(11)].

It can be seen that the oxygen atom in the hydroxyl group of monoethanolamine O (4) (-0.374 eV) exhibits more nucleophilic properties of the molecule. At the same time, the electrophilicity of the proton of the hydroxyl group in the H (11) molecule (0.199 eV) is high compared to other protons. This ensures the orientation of the acetylene molecule in the interaction with monoethanolamine towards the hydroxyl group and the replacement of the vinyl group by the hydrogen of the hydroxyl group.

Based on these theoretical quantum-chemical calculations, it was concluded that the main product of the vinylation processes in the work are O-vinyl ethers.

The process of vinylation of monoethanolamine using acetylene is carried out according to the following scheme.

$$NH_2-CH_2-CH_2-OH + CH\equiv CH \xrightarrow[^{o}t]{KOH/DMSO} NH_2-CH_2-CH_2-O-CH=CH_2$$

**Results.** In this work, we studied the influence of various factors on the vinylation of monoethanolamine in a KOH/DMSO medium in the presence of acetylene, the optimal synthesis conditions were determined. In the course of research, experiments were carried out at an acetylene pressure of 4 atm. The effect

of temperature and time on the vinylation reaction in the presence of 20% KOH catalyst relative to the mass of monoethanolamine was studied. The results obtained are presented in tabl. 1.

Table 1

**Temperature and reaction to the reaction of monoethanolamine with acetylene duration effect (the amount of KOH catalyst in relation to the mass of monoethanolamine is 20%)**

| Temperature, °C | Reaction time, hour | Output of vinyl ester, % |
|---|---|---|
| 80 | 5 | 38,3 |
| 80 | 6 | 48,6 |
| 80 | 7 | 50,1 |
| 85 | 5 | 49,7 |
| 85 | 6 | 51,0 |
| 85 | 7 | 51,3 |
| 90 | 5 | 53,5 |
| 90 | 6 | 54,6 |
| 90 | 7 | 53,1 |
| 95 | 5 | 50,2 |
| 95 | 6 | 51,3 |
| 95 | 7 | 50,5 |

**Discussions.** As can be seen from the results, temperature markedly affects the yield of monoethanolamine vinyl ether. When the temperature rises from 80°C to 95°C, the product yield increases from 38,3 to 54,6%. As a result of a further increase in temperature, the yield of the product decreased. This condition is explained by a decrease in the solubility of acetylene in a solvent at very high temperatures, as a result of which its concentration also decreases, the reaction rate and the yield of the resulting product decrease.

In addition, increasing the reaction time from 5 to 6 hours increases the product yield. However, by 7 o'clock the yield of the product is reduced. With a long course of the reaction at high temperatures, the formation of resinous oligomeric substances with vinyl esters and other reaction intermediates is observed with dimethyl sulfoxide [14-20].

The synthesized vinyl esters were purified and isolated by vacuum desorption and column chromatography. The purity of the product was determined by the GSX method, the elemental composition was calculated, and the structural formula was analyzed by IR spectra.

**Conclusion.** Monoethanolamine was found to form monoethanolamine o-vinyl ester with a maximum content of 54.6% when exposed to acetylene for 6 hours at 90° C. and 4 atm pressure in a high KOH/DMSO environment.

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# SOLUBILITY OF COMPONENTS IN THE SYSTEM $NaClO_3$ $CO(NH_2)_2$-$NH(C_2H_4OH)_2$ -$H_2O$

**MARDANOV SAIDOLIM**

Researcher of Fergana Politechnical Institute;
E- mail.: mardonov82@yandex.ru, Phone.: (+99894) 193-82-18

**KHAMDAMOVA SHOKHIDA**

Associate professor of Fergana Politechnical Institute
E- mail.: hamdamova79@mail.ru, Phone.: (+99891) 663-46-35

**Abstract**:
Solubility of components in $NaClO_3$ $CO(NH_2)_2$ - $NH(C_2H_4OH)_2$ -$H_2O$ from total freezing temperature (-67.4**°C**) to 40.0**°C** has been studied. A polyhermic solubility diagram has been constructed on which the areas of crystallization of ice, carbamide, sodium monocarbamidochlorate, and diethanolamine have been delimited. The system relates to a simple eutonic type.

**Keywords:** components, polyhermic, diagram, ice, carbamide, crystallization, sodium, monocarbamidochlorate, diethanolamine, temperature.

**Introduction.** The search and development of lowtoxic, highly effective and mild defoliants that do not adversely affect the yield of cotton, the technological performance of cotton fiber is an urgent problem in cotton growing.

Cotton growing is one of the most important branches of agriculture in Uzbekistan. In case of chemical impact on cotton in order to remove leaves, highly effective defoliants are needed, providing more than 80% fall of cotton leaves in one treatment at low consumption rates, acting “softly” on plants, and therefore not negatively affecting seed oil content, yield, quality cotton fiber and do not clog it [1, 2]. Meanwhile, the sodium chlorate produced in the republic and used as a cotton defoliant does not fully meet the modern requirements of cotton growing [3, 4]. The "rigidity" of its effect on plants requires the creation of new effective, mild defoliants for plants.

In this regard, special attention is paid to the production of highly effective, low-toxic and physiologically active defoliants. The existing chlorate-based defoliants do not meet modern requirements for defoliants. It is known that the defoliating effect of chlorates is always to some extent accompanied by a desiccation effect [5, 6].

When explaining the growth activity of ethanolamines, it should be taken into account that in the presence of carbon dioxide and oxygen, ethanolamines can form glycerol, glycol, oxalic, formic, naphthic, and acetic acids, which belong to the group of growth substances [7–8]

For successful defoliation of cotton, preparations are needed that provide a high degree of leaf fall and bolls opening. One of the possible ways to solve this important

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