SYNTHESIS OF ACETYLENE ALCOHOLS BASED ON THE ETHINYLATION OF SOME HETEROATOMIC ALDEHYDES WITH THE PARTICIPATION OF VARIOUS CATALYSTS

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Abstract: In this research work, the catalytic systems InBr3/Et3N/Et2O and ProPhenol/Me2Zn/TGF were used for the first time, based on the reactions of enantioselective ethinylation of acetylene, phenylacetylene with some aldehydes - thiophene-2-carbaldehyde, 3-methylthiophene-2-carbaldehyde, furan-2- carbaldehyde, pyridine-3-carbaldehyde, quinoline-2-carbaldehyde and 3-bromo-4-pyridinecarbaldehyde, the synthesis reactions of the corresponding acetylene alcohols were studied. The influence of the used complex systems on the efficiency of the formation of acetylene alcohols is proposed. and reaction mechanisms are presented. A number of factors influencing the yield of the product were studied - temperature, reaction duration, influence of the amount of catalyst and solvents, substrate and reagents. Based on the results obtained, the most alternative conditions for the processes were found. The composition, purity, structure and quantum chemical properties of the synthesized acetylene alcohols were confirmed by modern physicochemical methods - IR, ^{1}H-NMR, ^{13}C-NMR spectroscopy, mass spectrometry, chromatography. A number of production efficiencies of synthesized acetylene alcohols have been determined. In the process, a number of propensities for the nucleophilic addition reaction were determined in accordance with the nature of the substituents retained by the heteroatomic element in the aldehyde molecule, their spatial arrangement and nature of action.

Key words: aldehydes, alkynes, prophenol, indium (III) bromide, tetrahydrofuran, diethyl ether, acetylene alcohols, triethylamine, diethyl ether, reaction mechanism.

1 INTRODUCTION

The creation of technologies for the synthesis and production of acetylene alcohols by alkynylation reactions of organic compounds with a carbonyl group in their molecule using acetylene and its homologues by homogeneous or heterogeneous catalytic methods is being developed [1-3]. The molecule of acetylene alcohols has several reaction centers, and they are widely used as biologically active substances in the production of disinfectants, rodenticides, defoliants, solvents, antibiotics, hormones and dyes [4-5].

2 RESEARCH METHODOLOGY

The reaction was carried out in a reactor equipped with a 2000 ml four-neck flask made of heat-resistant transparent glass (Borosilicate glass 3.3, Metric 300 mm/29/32) with a reflux cooler (Brand allihn, Isolab, DIN 12581, 400 mm/29/32), dropper funnel (DURAN brand, Linz brand 250ml/29/32), thermometer (DIN, Mercury, 8-9 mm, brand -10/+100), mixer (EUROSTAR 20, 150-6000 rpm, 50-60 Hz brand). The composition, purity and structure of the synthesized acetylene alcohols were analyzed using ^{1}H, ^{13}C NMR spectra (Bruker Avance 400 and 100 MHz, at a temperature of 20-25 ºC, in the presence of $CDCl_3$ and C_6D_6 solvents).

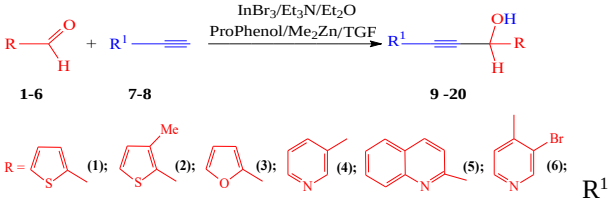
3 ANALYSIS OF WORKS RELATED TO THE TOPIC

The interest of scientists in acetylene alcohols makes it possible to find solutions to difficult situations in the chemical industry. In particular, propargyl alcohols were synthesized in high yield by carrying out enantioselective alkynylation reactions with the addition of carbonyl compounds of lithium acetylenides in the presence of a lithium binaphthalate catalyst [8]. In the synthesis of acetylene alcohols, the use of aldehydes with heteroatomic substituents in their molecules opens the way to increasing their biological activity [9]. Also, new types of catalytic systems are being developed for the synthesis of acetylene alcohols with high efficiency [10-11]. Chinese scientists reacted aliphatic aldehydes in a catalytic system prepared from a mixture of diethylzinc, xylene and benzene with phenylacetylene at

a temperature of -20 °C and synthesized aromatic acetylene alcohols in yields of 51-84%. [12]. Alkynylation of ketones and aldehydes was carried out with the component NaOH, KOH or CsOH/KO^{t}Bu/DMSO. [13]. As a result of the enantioselective alkynylation of aldehydes and ketones in the $Me_2Zn/Ti(O^iPr)_4/BaF_2$/toluene catalytic system, the synthesis of acetylene alcohols was achieved with a yield of 98%. [14]. Reactions of enantioselective nucleophilic addition of alkynes to ketones were carried out in the presence of organometallic reagents [15]. Diacetylene alcohol 3-methyl-1,5-diphenylpenta-2,4-dyne-3-ol, which has high activity for smallpox vaccination, was synthesized in the presence of a reaction of Iocich phenylethinylmagnibromide with ethyl acetate [16]. Secondary acetylene alcohols are obtained by the action of aldehydes in the presence of Ethyl Zinc and tetrahydrofuran on an ion-exchange acetylene compound [17]. Secondary propargyl alcohols with 46-67% were synthesized by ethinylation of aromatic and heteroatomic aldehydes with acetylene using the con-H_2O-DMSO catalytic system. This process was carried out at atmospheric pressure for 3 hours at temperatures of 5-7 °C. [18]. Using the Grignard reaction, the corresponding acetylene alcohols were synthesized in 57-85% yields by reacting R–CC–MgX compounds with carbonyl compounds (aldehydes, ketones) in a solution of tetrahydrofuran and diethyl ether. [19]. Aromatic aliphatic aldehydes react with phenylacetylene or hexine-1 at room temperature to form 1-(3-chlorophenyl)-3-phenylprop-2-yn-1-ol, 1-(2,4-chlororophenyl)-3-phenylprop-2-yn-1ol. The synthesis of propargyl alcohols in 98% concentration was carried out. [20]. The reaction of methylpropyl ketone, dimethyl ketone, methyl isopropyl ketone and pinocholines with phenylacetylene was carried out and acetylene alcohols were synthesized using organic magnesium compounds. The effect of diethyl ether and tetrahydrofuran solvents on the reaction yield was studied and a high yield was synthesized when the process was carried out in a tetrahydrofuran solution. [21].

4 ANALYSIS AND RESULTS

In this work, for the first time, the synthesis reactions of acetylene alcohols - 1-(thiophenyl-2)prop-2-yn-1-ol, 1-(3-methylthiophenyl-2)prop-2-yn-1-ol, 1-(furanyl-2)prop-2-yn-1ol, 1-(pyridinyl-3)prop-2-yn-1-ol, 1-(quinolinyl-2)prop-2-yn-1-ol, 1-(3-bromopyridinyl-4)prop-2-yn-1ol, 1-(thiophenyl-2)-3-phenylprop-2-yn-1-ol, 1-(3-methylthiophenyl-2)-3-phenylprop-2-yn-1-ol, 1-(furanyl-2)- 3-phenylprop-2-yn-1-ol, 1-(pyridinyl-3)-3-phenylprop-2-yn-1-ol, 1-(quinolinyl-2) phenylprop-2-yn-1-ol, 1-(3-bromopyridinyl-4) phenylpropinprop-2-yn-1-ol, were carried out by alkynylation reaction of aldehydes containing in their molecule sulfur, oxygen, nitrogen and bromine - thiophene-2-carbaldehyde, 3-methylthiophene-2-carbaldehyde, furan-2-carbaldehyde, pyridine-3-carbaldehyde, quinoline-2-carbaldehyde and 3-bromo-4-pyridinecarbaldehyde with acetylene and phenylacetylene using $InBr_3/Et_3N/Et_2O$ and ProPhenol/Me_2Zn/TGF catalytic systems. Based on literature sources, the following reaction scheme of the process is proposed.



R^1 = H (7); C_6H_5 (8);

The influence of reaction duration, temperature, molar amount of starting substances, the nature of solvents and catalysts on the course of the process and the yield of acetylene alcohols in selected catalytic systems was studied [22-23]. In particular, the most alternative conditions for the selected catalysts for the reaction of nucleophilic coupling of aldehydes with acetylene and phenylacetylene, which are the object of study, were determined. In the $InBr_3/Et_3N/Et_2O$ catalytic system, the starting materials in the process are in an equimolar ratio, the temperature is 20 °C, the reaction duration is 120 minutes, the product yields are 9-72%, 10-77%. 11-92%, 12-84%, 13-79%, 14-89%, 15-69%, 16-73%, 17-89%, 18-78%, 19-75%, 20-85% were synthesized with the highest yields. While in the ProPhenol/Me_2Zn/TGF catalytic system, the starting substances and the number of moles in the catalytic system are in the ratio 1:1.25:0.25:1 mol, the solvent tetrahydrofuran, at a temperature of -5-0 ° C for 24 hours, the yield of heteroatomic acetylene alcohols is based on an increase in the number of collisions of molecules and ions in the system consists of 9-70%, 10-73%, 11-89%, 12-81%. 13-77%, 14-88%, 15-64%, 16-67%, 17-87%, 18-75%, 19-72%, 20-80% it is synthesized with the highest yield. In the process, the most alternative conditions were determined for the selected $InBr_3/Et_3N/Et_2O$ and ProPhenol/Me_2Zn/TGF catalytic systems and compared to the ProPhenol/Me_2Zn/TGF catalytic system, the product yield in the $InBr_3/Et_3N/Et_2O$ catalytic system was relatively efficient. In this case, $InBr_3$ forms an acetylenide that easily attacks the highly active carbonyl group while forming a basic environment in the system, lowering the activation energy of the reaction. was

observed to increase the selectivity of the nucleophilic coupling reaction. In addition, as a result of the rapid exchange of alcoholate and hydrogen chloride formed in the process, favorable catalytic conditions are created for the formation of acetylene alcohols.

Depending on the nature of the substituents retained by the heteroatomic element in the aldehyde molecule selected for the process carried out in various catalytic systems, their spatial location and nature of action, a number of propensities to the nucleophilic addition reaction are as follows: thiophene-2-carbaldehyde < 3-methylthiophene-2-carbaldehyde < quinoline-2-carbaldehyde < pyridine-3-carbaldehyde < 3-bromo-4-pyridine carbaldehyde < furan-2-carbaldehyde has been found to increase.

The composition, purity and structure of the synthesized acetylene alcohols were analyzed using ^{1}H and ^{13}C NMR spectra.

1-(thiophenyl-2)prop-2-yn-1-ol (**9**) – R_f= 0.68; (72%), ^{1}H NMR:: δ 7.33 (dd, 1H, $H_{5,Th}$), 7.19 (dd, 1H, $H_{4,Th}$), 6,92 (m, 1H, $H_{3,Th}$), 5.61 (s, 1H), 2.64 (s, 1H, C≡CH), 2.38 (d, 1H, OH); ^{13}C NMR:: δ 145.7, 127.8, 127.3, 126.8, 85.6, 70.1, 61.5.

1-(3-methylthiophenyl-2)prop-2-yn-1-ol (**10**) – R_f= 0.57; (77%), ^{1}H NMR:: δ 7.64 (dd, 1H, $H_{4,Th}$), 7.22 (dd, 1H, $H_{3,Th}$), 5.43 (d, 1H), 2.71 (s, 1H, C≡CH), 2.29 (d, 1H, OH), 1.86 (s, 3H, CH_3); ^{13}C NMR: δ 141.7, 132.8, 127.4, 124.6, 84.3, 72.6, 65.7, 12.8.

1-(furanyl-2)prop-2-yn-1ol (**11**) – R_f= 0.68; (92%), ^{1}H NMR: δ 6.92 (m, 1H, CH_{Fur}), 6.14 (m, 1H, CH_{Fur}), 5.87 (m, 1H, CH_{Fur}), 5.08 (s, 1H), 2.36 (m, 1H, C≡CH), 2.02 (m, 1H, OH); ^{13}C NMR: δ 152.7, 142.6, 113.5, 106.8, 81.9, 71.4, 57.6.

1-(pyridinyl-3)prop-2-yn-1-ol (**12**) – R_f= 0.47; (84%), ^{1}H: δ 8.44 (d, 2H, $2CH_{Pir}$), 7.64 (m, 1H, CH_{Pir}), 7.52 (s, 1H, CH_{Pir}), 5.16 (s, 1H), 2.45 (s, 1H, C≡CH), 2.09 (d, 1H, OH); ^{13}C NMR: δ 155.6, 149.9, 137.2, 136.8, 121.7, 87.4, 72.6, 63.3.

1-(quinolinyl-2)prop-2-yn-1-ol (**13**) – R_f= 0.47; (79%), ^{1}H NMR: δ 8.27 (d, 1H, CH_{Xin}), 8.15 (d, 1H, CH_{Xin}), 7.96 (m, 1H, CH_{Xin}), 7.48 (m, 2H, $2CH_{Xin}$), 7,25 (m, 1H, CH_{Xin}), 5,63 (m, 1H), 2.93 (s, 1H, C≡CH), 2.26 (d, 1H, OH); ^{13}C NMR: δ 159.7, 146.4, 136.2, 128.3, 127.4, 126.5, 124.9, 121.8, 83.7, 70.9, 66.9.

1-(3-bromopyridinyl-4)prop-2-yn-1ol (**14**) – R_f= 0.57; (89%), ^{1}H NMR: δ 8.34 (s, 1H, CH_{Pir}), 8.17 (m, 1H, CH_{Xin}), 7.48 (d, 1H, CH_{Pir}), 5,56 (m, 1H), 2.94 (s, 1H, C≡CH), 2.15 (s, 1H, OH); ^{13}C NMR: δ 149.5, 147.6, 144.2, 127.3, 123.1, 84.7, 70.6, 61.8.

1-(thiophenyl-2)-3-phenylprop-2-yn-1-ol (**15**) – R_f= 0.33; (69%), ^{1}H NMR: δ 8.12 (m, 2H, $2CH_{Th}$), 7.57 (m, 5H, $5CH_{Ph}$), 7.24 (m, 1H, CH_{Th}), 5,89 (d, 1H), 2.34 (d, 1H, OH); ^{13}C NMR: δ 149.3, 129.6, 128.1, 127.0, 126.3, 121.5, 88.9, 84.7, 64.9.

1-(3-methylthiophenyl-2)-3-phenylprop-2-yn-1-ol (**16**) – R_f= 0.37; (73%), ^{1}H NMR: δ 7.69 (m, 2H, $2CH_{Ph}$), 7.35 (m, 5H, $3CH_{Ph,}$ $2CH_{Th}$), 5.62 (d, 1H), 2.23 (d, 1H, OH), 1.96 (s, 3H, CH_3); ^{13}C NMR: δ 142.7, 131.3, 128.3, 127.2, 125.9, 122.1, 89.6, 85.8, 63.2.

1-(furanyl-2)- 3-phenylprop-2-yn-1-ol (**17**) – R_f= 0.41; (89%), ^{1}H NMR: δ 7.46 (m, 3H, $3CH_{Ph}$), 7.25 (m, 3H, $2CH_{Ph,}$ CH_F), 6.42 (m, 1H, CH_F), 6.29 (m, 1H, CH_F), 5.26 (d, 1H), 1.97 (d, 1H, OH); ^{13}C NMR: δ 154.2, 144.3, 129.6, 127.8, 121.7, 112.2, 107.5, 89.6, 84.4, 66.9.

1-(pyridinyl-3)-3-phenylprop-2-yn-1-ol (**18**) – R_f= 0.33; (78%), ^{1}H NMR: δ 8.44 (m, 2H, $2CH_{Pir}$), 7.59 (m, 2H, $2CH_{Pir}$), 7.41 (m, 2H, $2CH_{Ph}$), 7.33 (m, 3H, $3CH_{Ph}$), 5.74 (d, 1H), 2.86 (d, 1H, OH); ^{13}C NMR: δ 152.6, 146.7, 134.2, 132.1, 129.8, 127.3, 121.5, 86.9, 83.7, 60.4.

1-(quinolinyl-2) phenylprop-2-yn-1-ol (**19**) – R_f= 0.47; (75%), ^{1}H NMR: δ 8.27 (d, 1H, CH_{Naphth}), 8.16 (m, 3H, $3CH_{Naphth}$), 7.68 (m, 2H, $2CH_{Naphth}$), 7.37 (m, 2H, $2CH_{Ph}$), 7.18 (m, 3H, $3CH_{Ph}$), 5.44 (d, 1H), 2.69 (d, 1H, OH); ^{13}C NMR: δ 159.4, 148.7, 136.5, 129.4, 127.9, 126.3, 121.6, 89.8, 84.6, 65.3.

1-(3-bromopyridinyl-4) phenylpropinprop-2-yn-1-ol (**20**) – R_f= 0.31; (85%), ^{1}H NMR: δ 8.52 (m, 2H, CH_{Pir}), 7.76 (s, 1H, CH_{Pir}), 7.46 (m, 2H, $2CH_{Ph}$), 7.14 (m, 3H, $3CH_{Ph}$), 5.23 (d, 1H), 2.18 (d, 1H, OH); ^{13}C NMR: δ 152.6, 147.5, 127.8, 126.2, 121.8, 120.4, 88.3, 85.6, 57.4.

CONCLUSION AND RECOMMENDATIONS

For the first time, acetylene alcohols were synthesized in $InBr_3/Et_3N/Et_2O$ and ProPhenol/Me_2Zn/TGF catalytic systems. The most alternative conditions for alkynylation processes of selected aldehydes were found. Regimes for managing and monitoring alkynylation processes have been determined, reaction mechanisms have been proposed, kinetic changes have been determined, and the structure, composition and purity of the synthesized compounds have been proven using modern research methods. The effect of heteroatoms in the aldehyde molecule on product formation was studied.

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