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Synthesis of 2-Methilgexin-3-Diol-2,5 by the Heterogenative-Catalytic Method

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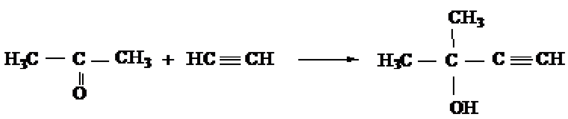
ABSTRACT: The article presents the results of a study of heterogeneous-catalytic interaction of acetylene and acetone with the formation of 2-methylhexin-3-diol-2.5 using copper-cadmium-zinc-kaolinic (CCZK) and copper-cadmium-zinc-bentonite (CCZB) catalysts in depending on their nature, content and temperature of the reactions.

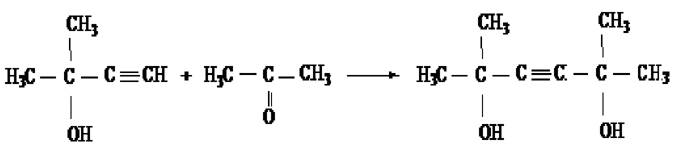
KEYWORDS:2-methylhexin-3-diol-2.5, heterogeneous catalysis, acetylene, acetone, copper-cadmium-zinc-kaolin, copper-cadmium-zinc-bentonite, temperatures.

I. INTRODUCTION

Acetylenic alcohols are widely used for the synthesis of various polymers and copolymers, as binding agents in the synthesis of artificial fibers, as well as in the form of defoliants and pesticides in agriculture, in addition they are the initial products in the preparation of glue, paints and plasticizers. As tanning components are used in the leather and textile industry, as well as in the form of raw materials in obtaining medical products.[1, 2].
The reactivity of acetylene and its derivatives is very high. In acetylene chemistry, new methods are being developed for the formation of various hetero and carboso compounds. The triple bond and the hydroxyl group in the molecule of acetylene alcohols greatly improve their chemical properties and expand the scope.
It is known that diols are synthesized on the basis of various reactions (by the methods of Favorsky, Iozic, Reppe) from carbonyl and acetylene compounds. At the same time, acetylenic alcohols are first formed, then acetylene glycols. As is known, in atoms of copper, silver, mercury, zinc, cadmium, nickel there are d-orbits, which form π-complexes with acetylene. Therefore, their compounds as a catalyst are widely used in the chemistry of acetylene.
The mechanism of formation of acetylene alcohols:



Formed 2-methylbutin-3-ol-2, joining another acetone molecule, forms 2,5-dimethylhexin-3-diol-2,5



The synthesis of acetylene alcohols by the homogeneous catalytic method, as well as the study of their properties, is given in some sources [3–9]. But their synthesis and research using the heterogeneous catalytic method has been little studied.
The formation of acetylene alcohols and acetylene glycols depends on the following factors: the nature and amount of the catalyst, the nature of the peptizers, the reaction medium, the feed rate of acetylene. High temperature in a certain range and low consumption of acetylene provide an increase in the efficiency of formation of acetylene glycols.

II. MATERIALS AND METHODS

100 g of the prepared sample of catalyst are placed in the reactor. Acetylene is blown from the gas line to the reactor, then 500 ml of acetaldehyde and acetone are added dropwise. The experiment is carried out at 135 ° C. After reaction, additional gases are removed from the reaction medium using a stream of nitrogen. Then the reaction mixture is left alone for 12 hours. The organic part is separated from the obtained catalyzate with icy distilled water, the aqueous portion is extracted 3-4 times with diethyl ether, filtered and distilled. Drying the catalyzate is carried out using $CaCl_2$.

III. RESULTS AND DISCUSSION

The influence of various factors on the synthesis of 2-methylhexin-3-diol-2.5, such as the nature and amount of the catalyst, temperature, reaction time, was studied. As a result of the interaction of acetylene with acetone, heterogeneous-catalytic synthesis of 2-methylhexin-3-diol-2.5 was carried out at a pressure of 0.1-0.5 MPa in the presence of catalysts: copper-cadmium-zinc-kaolin (CCZK), copper-cadmium-zinc-bentonite (CCZB), copper-cadmium-zinc-zeolite (CCZZ), copper-cadmium-zinc-silica gel (CCZS).

It is established that an increase in the amount of the active component of copper oxide in the composition of the catalyst significantly affects the process.

And the oxides of cadmium, bismuth and zinc increase the lifetime of the catalyst, thereby reducing the processes of oligomerization, carbonization, vinylization, polymerization. This is explained by the fact that the coordination ability of copper is greater than that of other metals; therefore, π-complexes with acetylene triple bonds are formed.

In the case of using copper-cadmium-zinc-kaolin catalyst, the product yield is 84.7%.

Thus, the catalytic actions of CCZK are more active than those of other catalysts.

Some parameters of the catalysts are given in table 1.

Table 1

The effect of the nature of the catalyst on the synthesis of 2-methylhexin-3-diol-2.5 (peptizer HNO_3, NH_4OH, carboxymethylcellulose, temperature 135°C)

Catalyzer	Contents of catalyzer (%)	Product yield, %
CCZK	CuO-40; CdO- 8, ZnO-2, Kaolin-50	84,7
CCVK	CuO-40, CdO - 8, Bi_2O_3 -2, Kaolin-50	80,7
CCZB	CuO-40; CdO- 8, ZnO-2,, Bentonite-50	82,1
CCVB	CuO-40, CdO - 8, Bi_2O_3 -2, Bentonite-50	77,6
CCZS	CuO-40; CdO- 8, ZnO-2, silica gel-50	79,5
CCZS	CuO-40, CdO - 8, Bi_2O_3 -2, silica gel-50	76,7
CCZZ	CuO-40; CdO- 8, ZnO-2, zeolite-50	75,9
CCVZ	CuO-40, CdO - 8, Bi_2O_3 -2, zeolite-50	71,8

Analysis of the research results show that catalysts, including kaolin, have a greater effect on the yield of the main product than with bentonite, silica gel and zeolite. This is due to the fact that kaolin contains oxides Cr_2O_3, Fe_2O_3, FeO, which play the role of a promoter enhancing the catalytic activity of the catalyst.

The work also studied the effect of the nature of peptizers on the product yield.

HNO_3, NH_4OH, carboxymethylcellulose, CH_3COOH, sodium polygalacturonate, sodium polyacrylate were used as peptizers, the results of which are shown in Fig.1.

Analysis of the research results (Fig. 1) shows that the maximum value is obtained when using carbrimetsecellulose, HNO_3, NH_4OH as a peptizer.

This is due to the large binding capacity, leading to strength, porosity and a large contact surface. It is evident from Fig. 1 that with an increase in the duration of time, the yield of products decreases, which is explained by the formation of resinous substances, such as oligomers and polymers, and this in turn leads to a decrease in the main product.

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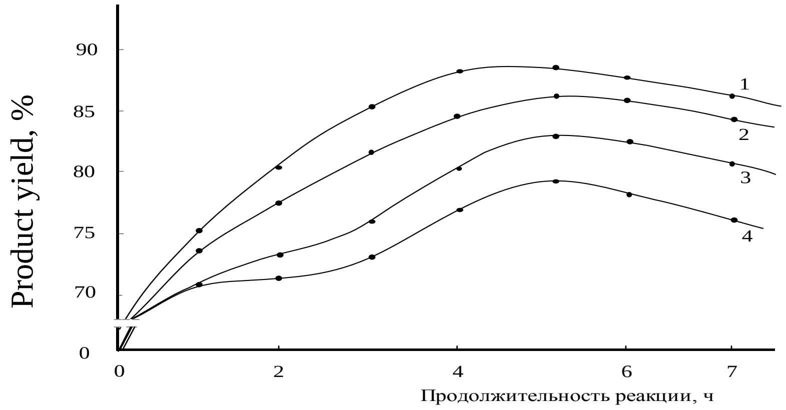

Fig.1. Influence of the nature of peptizers on the product yield

1 – HNO_3, NH_4OH, carboxymethylcellulose; 2 – HNO_3, NH_4OH, CH_3COOH; 3 – HNO_3, NH_4OH, sodium polygalacturonate; 4 – HNO_3, NH_4OH, sodium polyacrylate

In addition, the effect of temperature, which is one of the most important factors influencing the product yield, was studied. The effect of temperature was studied in the range of 105-145ºC. The results are shown in table 2.

The analysis of the results obtained shows that with increasing temperature the product yield increases, the optimum temperature for the process is 135°C. A further increase in temperature leads to a decrease in the yield of the product; this is due to the fact that at high temperatures, due to the triple bonds, the synthesized substance undergoes an oligomerization and polymerization reaction.

The dependence of the product yield on the amount of the active component of copper oxide contained in the catalyst has also been studied.

Table2
The effect of temperature on product yield

Catalyzer	Temperature, °C	Product yield,%
CCZK	105	74,9
	115	78,5
	125	84,4
	135	88,4
	145	85,6
CCZB	105	72,4
	115	76,5
	125	81,3
	135	86,1
	145	81,8
CCZS	105	69,7
	115	73,4
	125	79,5
	135	83,5
	145	80,3
CCZZ	105	66,5
	115	70,3
	125	75,7
	135	79,8
	145	76,4

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Analysis of the research results shows that the product yield will be maximum when the amount of copper oxide increases to 40%, then the product yield decreases, this is because chemical bonds can be formed with the functional groups of the metal compounds (Cu, Cd), which negatively affect product yield.

The individual features of the synthesized 2-methylhexin-3-diol-2.5 are characterized by IR spectra. In the IR spectrum, absorption bands are observed in the region of 3700–3500 cm^{-1}, related to the deformation vibration of hydroxyl groups, and stretching vibrations of the same group are manifested in the region of 1400 cm^{-1}. For methylene groups, the absorption bands are observed in the region of 2850 cm^{-1}, the deformation vibrations of the corresponding triple bond –C≡C- in the region of 2500 cm^{-1}, and also the vibration for the C – O group in the region of 1110 cm^{-1}.

Thus, the results of IR spectroscopy confirm the structure of 2-methylhexin-3-diol-2.5.

IV. CONCLUSION

Synthesized 2-methylhexin-3-diol-2.5 using a heterogeneous-catalytic method based on acetylene, acetic aldehyde and acetone. The influence on the synthesis process of the nature of the catalyst, the amount of carrier and peptizer used in the preparation of the catalyst, the number of active components, temperature and reaction time is studied.

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