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Joint Synthesis Of Acetone And Acetaldehyde From Acetylene

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ABSTRACT

The study investigated the possibility of using catalysts based on compounds of cadmium, zinc and other elements filled with orbital electrons d10, having several polyfunctional properties, as well as promoted by compounds of various metals in the hydration reaction of acetylene and its derivatives. The acetylene hydration reaction yields acetaldehyde or acetone depending on the composition and nature of the catalyst used under the same conditions. In particular, the formation of acetylene at 360-440 0C in the presence of a catalyst from cadmium fluoride-containing 20% CdF2, 5.0% AlF3, 75% Al2O3: acetaldehyde formation when reacted with water = 1: 3-1: 5, cadmium fluoride, It has been found that using catalysts containing zinc, iron, nickel, cobalt compounds instead of aluminium fluoride, the process leads to the formation of acetone.

KEYWORDS

Acetylene, acetone, acetaldehyde, catalyst, selectivity, kinetics, mechanism.

INTRODUCTION

It is known that acetone is used in the manufacture of paints, synthetic fibres, films, black powder. It is also used to increase the octane number of motor fuels, in organic synthesis and as a solvent [1-2].

One of the main tasks of chemists is to create new methods and technologies for the production of substances that are important for the national economy, which can replace imported products

using local raw materials and exhaust gases, and on this basis to increase the country's export potential. In this regard, it is important to create catalysts for the synthesis of acetone [3-16], acetaldehyde [17-18], vinyl acetate [19-24] and vinyl chloride [25], the preparation of which is cheap and simple, and to accelerate the process. An effective solution to this problem is to create an active catalyst with high selectivity and efficiency, which is an urgent task of this research work. The annual demand for vinyl acetate is 15,000 tons, the annual demand for vinyl chloride and chloroprene is 30,000 tons, and the annual demand for acetone is 10,000 tons. Today, Navoi Azot produces 30,000 tons of acetylene per year by pyrolysis. Currently, acetone is obtained in 2 ways: cumene method and isopropyl alcohol [26-27]. The absence of kumol and isopropyl alcohol in the country does not allow to obtain acetone by these methods. The most alternative, cost-effective way to obtain acetone in our country is the direct catalytic hydration of acetylene. Although this method of obtaining acetone from acetylene is known in the literature, it has not been implemented on an industrial scale due to the lack of stable, active, and selective catalysts. Among the hydration reactions of acetylene compounds, the reaction of their hydration to acetaldehyde using a cadmium-calcium phosphate catalyst has been studied in detail and introduced into the industry [1-2].

However, the cadmium-calcium phosphate catalyst used in industry has several disadvantages: the conversion of acetylene is low, the selectivity is low, the yield is not high, the thermally unstable, and so on. The work aims to study the catalytic properties of various catalysts and to create new original catalysts for the production of acetone and acetaldehyde based on local raw materials and catalytic hydration of acetylene in exhaust gases.

EXPERIMENTAL PART

Experiments on the catalytic hydration of acetylene and its derivatives in the gas phase were carried out on a reactor 25 mm in diameter and 1000 mm in height, made of stainless steel under stationary conditions. The qualitative and quantitative composition of the reaction products was studied by gas-liquid chromatography under the following conditions: stationary phase 15% Apiezon-M in Cvetochrome, column thermostat temperature 80 0C, carrier gas flow rate - helium 60 cm3/min, detector-DIP. Quantitative analysis was carried out by the method of internal standards. Specific surface area, crushing strength, total pore volume and ash content of the samples were determined.

The specific surface area was determined by the method of thermal desorption of nitrogen in a flow of a carrier gas - helium at the boiling point of liquid nitrogen; the experimental data were processed using the BET equation.

The phase composition of the samples was determined by the method of X-ray diffractometric, the shooting was carried out on a DRON-3M diffract meter on CuKα-radiation with a Ni-filter, the length of the X-ray radiation. The specific surface area of the obtained catalyst was calculated by the BET method. Dispersed properties of the catalyst were studied using a scanning electron microscope (JSM-6510 LV). The catalytic activity of the obtained sample was studied using the acetylene hydration reaction.

Acetaldehyde and acetone were synthesized as follows. Acetylene was saturated with water at a temperature of 70-80 0C and a ratio of water: acetylene = (1: 3) - (1: 5) mol was passed through the catalyst bed at 360 0C with a space velocity of 180-200 h-1. The vapour-gas mixture leaving the reactor was cooled in a refrigerator. The reaction products were trapped in water. The catalyst contains acetaldehyde, acetone, crotonaldehyde, paraldehyde, etc. To maintain the degree of acetylene conversion at least 80%, the reaction temperature was raised by 100C every 20 h. After 96-120 hours, the degree of conversion of acetylene decreases to 75-70%. Then the reaction was stopped and the catalyst was regenerated in a known manner. Subsequently, the effect of various

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parameters (temperature, space velocity, acetylene-water ratio) on the acetylene conversion and acetone yield was studied.

As a result of studying the effect of temperature on the yield of acetone, it was found that in the temperature range of 360-500 0C the dependence between the reaction yield and temperature is extreme and at 450 0C the yield was considered maximum.

EXPERIMENTAL RESULTS AND THEIR DISCUSSION

We explored the possibility of using catalysts based on compounds of cadmium, zinc and other elements filled with *d10* orbital electrons with several polyfunctional properties, as well as promoted by compounds of various metals in the hydration reaction of acetylene and its derivatives. During the promotion of catalysts prepared by suspension on the basis of cadmium and zinc fluorides and other compounds, the formation of hydroxyl-fluorides with the following composition:

Cd(OH)F, Zn(OH)F, Al(OH)2F, Al(OH)F2 studied by the analytical method and found that the formation of hydro fluorides leads to an increase in catalyst activity. The increase in catalyst activity is explained as follows: when the catalyst is in the polymorphic-crystalline state, it retains the bound water molecules and therefore has high catalytic activity. As the temperature increases from 500 to 650 0C, the proportion of the crystalline phase increases, the amount of chemically bound water decreases, and the hydroxy fluorides begin to decompose. As a result, its activity decreases due to the decrease in the specific surface area of the catalyst. More than 10 catalysts were tested for the catalytic hydration reaction of acetylene. The result was a new cadmium-fluorine-aluminium catalyst promoted by zinc fluoride and aluminium fluorides for the catalytic hydration reaction of acetylene and its derivatives (Table 1).

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Table 1.

Composition and properties of synthesized cadmium- fluorine-aluminum catalysts (t = 723 k).

Catalyst composition, mass%	Specific surface area, m^2 / g	Operating time before regeneration, daily	Productivity, g/kg, cat • h	Conversion of acetylene, %
CdF2 - 20 Al2O3 -80	220	96	110	95
CdF2 - 15 Al2O3 - 85	165	120	85	82
CdF2 - 11,2 AlF3 - 2,8 Al2O3 - 86	135	140	91	90
CdF2 - 15 AlF3 - 5 Al2O3 - 80	151	155	100	92
CdF2 - 10 AlF3 - 10 Al2O3 - 80	183	130	88	94
CdF2 - 5 ZnF2 - 10 AlF3 - 5 Al2O3 - 80	201	160	84	86

As can be seen from Table 1, the given catalysts provide high acetylene conversion. Their stability is 1.5-2.0 times higher than that of cadmium-calcium-phosphate catalyst. The catalyst promoted by up to 5.0% aluminium fluoride has sufficient activity, mechanical strength and stability. The catalyst, which contains 10% to 20% cadmium fluoride, provides high acetaldehyde yields. When the reaction is carried out at 300-420 0C, the yield of acetaldehyde is 90% higher than that of acetylene, which reacts. Additional products include acetone, 3- oxy-butanol, crotonaldehyde, butanol, ethyl acetate and paraldehyde.

The acetylene hydration reaction yields acetaldehyde or acetone depending on the composition and nature of the catalyst used under the same conditions. In particular, in the presence of a catalyst based on cadmium fluoride-containing 20 % CdF2, 5,0 % AlF3, 75 % Al2O3, acetylene reacts at temperature 360-440 0C in the ratio: water = 1:3 - 1:5. The influence of various factors (temperature, space velocity, acetylene: water ratio) on acetylene conversion and acetaldehyde yield was studied. A study of the effect of temperature on acetaldehyde output showed that the relationship between reaction yield and temperature in the range 360-440 0C is extreme, and the yield at 400 0C is maximum. The temperature is

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raised by 10 0C every 15-20 hours to keep the acetylene conversion constant at 80-85%. The cadmium-fluorine-aluminium catalyst used in this process was found to maintain its catalytic activity unchanged for 96-120 hours. The catalyst used in the reaction, which has lost its activity, resumes its initial activity after regeneration. When cadmium fluoride and aluminium fluoride are replaced by catalysts containing zinc, iron, nickel, and cobalt, the process leads to the formation of acetone. Table 2 below shows the effect of the active components of the catalyst on the yield of the reaction products.

Table 2.
Influence of catalyst composition on the reaction product yield.

№	Catalyst content, mass %	Acetylene conversion, %	Catalyst content, %	
			Acetaldehyde	Acetone
1	ZnO-15.0 CdO-5.0 Al_2O_3-80.0	86.0	12.0	67.0
2	ZnO-10.0 CdF_2-5.0 Al_2O_3-85.0	83.0	21.0	56.0
3	CdF_2-20.0 Al_2O_3-80.0	84.0	81.0	12.0
4	ZnO-15.0 ZnF_2-5.0 CdF_2-2.0 Al_2O_3-78.0	92.0	14.0	78.5
5	Ni_2O_3-10.0 Fe_3O_4-20.0 Al_2O_3-70.0	95.4	8.0	92.8

Table 2 shows that the yield of acetone in the presence of № 5 catalysts containing iron and nickel oxides is 92.8%, and the conversion of acetylene is 95.4%. When studying the effect of temperature on the yield of acetone in the presence of the newly created high-activity and high- performance № 5th catalyst, it was found that the optimum temperature for the acetylene hydration reaction is 450 0C.

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Table 3
Effect of volumetric velocity on acetaldehyde yield and acetylene conversion

Dimensional speed 1 -1 h^{-1}	Rotation rate of acetylene, %		S,
	General	Until acetaldehyde	%
80	98.0	30.2	30.8
100	92.5	48.5	52.4
120	88.9	82.0	92.2
140	80.0	72.0	90.0
160	74.5	64.0	85.9
180	68.3	52.8	77.3
200	60.6	45.6	75.2
220	50.5	36.2	71.7

The effect of temperature, volumetric velocity, acetylene: water ratio, etc. on acetaldehyde yield was studied. Acetylene: The catalyst stability was found to be 90 hours at a water-to-water ratio of 1: 3, and the maximum acetylene conversion was 80%. Acetylene: When the water-to-water ratio is 1: 5, the catalyst is continuously active for more than 140 hours [37-33]. At this time, the conversion of acetylene is 90%. When the effect of temperature was studied in the range of 300-440 0C, an increase in acetylene conversion and a decrease in acetaldehyde selectivity were found with increasing temperature. The study of the effect of temperature on acetaldehyde yield showed that the relationship between the reaction yield and temperature in the range of 360-440 0C is extreme and the yield has a maximum value at 400 0C. To keep the acetylene conversion constant at 80-85%, the temperature is raised to 10 0C for 15-20 hours. The influence of temperature, catalyst size, reactor parameters and catalyst bed height on the technological parameters of the process was studied. The effect of the height of the catalyst layer on the conversion rate of acetylene is shown in Figure 1.

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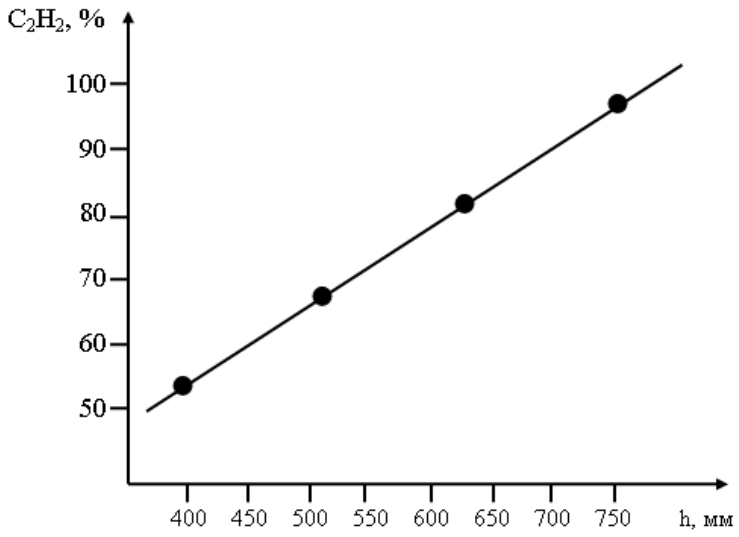

Figure 1. Effect of catalyst layer height on acetylene conversion rate

As shown in Figure 1, the acetylene conversion increases with increasing catalyst layer height, indicating that the reaction proceeds in the internal diffusion field. The ratio of the height of the catalyst layer to the diameter of the reactor is 50-60, and the volumetric velocity of acetylene is 50-60 h-1. The effect of temperature on acetone formation yield and acetylene conversion is shown in Table 4.

The table shows that the yield of acetone increases, the selectivity increases to 698 K. As the temperature rises above 698 K, the yield of acetone and the selectivity of the process decrease as the acetone is converted to other substances. It is known from the literature that cadmium and its compounds have a negative effect on the human body.

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Table 4.
Effect of temperature on acetone yield and acetylene conversion.
(C_2H_2: H_2O = 1:5 mol, kat: №5)

Harorat, ^0C	Asetonning hosil bo'lish unumi, %	Asetilen konversiyasi, %	Selektivlik, S %
350	47.0	62.0	75.8
375	59.8	75.0	79.7
400	70.9	85.0	83.4
425	84.8	90.0	94.2
450	65.0	94.0	63.8
500	52.0	97.8	53.2

Cadmium compounds are highly toxic (PDK 0.1 mg / m3). Therefore, we investigated the catalytic activity of cadmium-free compounds in acetylene hydration reactions.

Kinetics and mechanism of the catalytic hydration reaction of acetylene.

To create a scientific basis for the process of obtaining acetone by hydration of acetylene, it is necessary to study the kinetic laws of the reaction in the selected catalyst and to provide diagrams of the mechanism of the processes based on them.

It is known from the scientific literature that the kinetic laws of this process have been little studied in the presence of a small number of catalysts and there is no single view on the mechanism of the process. Based on this, several experiments were performed to study the kinetic laws of the catalytic hydration reaction of acetylene. To study the kinetic laws, the effect of the partial pressures of water and acetylene on the rate of acetone formation at a temperature in the range of 400-475 0C and a volumetric velocity (in acetylene) of 120 h-1 was studied.

The effect of the partial pressures of the reagents on the kinetic laws of the process was carried out by changing the partial pressure of one reagent while keeping the partial pressures of the remaining reagents constant. In order to keep the linear velocity of the initial mixture constant, the required amount of pure argon gas was sent to the reaction zone (field). The volume of the catalyst was adjusted accordingly to maintain a constant rate of acetylene flow. A study of the effect of the partial pressures of acetylene and water on the kinetic laws of the reaction found an increase in acetone yield with a decrease in the partial pressure of acetylene. At this time, the total conversion of acetylene increases,

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while the selectivity of the reaction relative to acetone decreases.

It is known from the literature that the interaction of polar acetylene molecules with water molecules leads to the formation of unstable vinyl alcohol, which isomerizes into acetaldehyde. The formed acetaldehyde first reacts with aldol, and then with croton to form 3-oximoid aldehyde or crotonic aldehyde. 3-oxime and crotonic aldehydes react with water to form hydrates. Based on the results of the experiments, the qualitative and quantitative composition of the reaction products, we propose the following mechanism for the formation of acetone in the presence of this catalyst:

$$CH \equiv CH + H_2O \rightarrow CH_3CHO$$
$$2CH_3CHO \rightarrow CH_3CH(OH)CH_2CHO$$

Hydration of 3-oxybutynin produces trihydric alcohol and dehydrogenation of the resulting product produces acetoacetic acid:

$$CH_3CH(OH)CH_2CHO + H_2O \rightarrow CH_3CH(OH)CH_2CH(OH)_2$$
$$CH_3CH(OH)CH_2CH(OH)_2 \rightarrow CH_3COCH_2COOH + 2H_2$$

Acetoacetic acid, in turn, decarboxylases under conditions to form acetone:

$$CH_3COCH_2COOH \rightarrow CH_3COCH_3 + CO_2$$

The above method of obtaining acetone is a promising method for Uzbekistan.

CONCLUSION

The possibility of using catalysts based on compounds of cadmium, zinc and other elements filled with d10 orbital electrons with several polyfunctional properties, as well as promoted by compounds of various metals in the hydration reaction of acetylene and its derivatives was studied. The hydration reaction of acetylene produces acetaldehyde or acetone, depending on the composition and nature of the catalyst used under the same conditions. In particular, the formation of acetylene at 360-4400S in the presence of cadmium fluoride catalyst containing 20 % CdF2, 5,0 % AlF3, 75 % Al2O3 formation of acetaldehyde when reacting with water = 1: 3 - 1: 5, cadmium fluoride, it was found that when catalysts containing zinc, iron, nickel, cobalt compounds are used instead of aluminium fluoride, the process leads to the formation of acetone.

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