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INFLUENCE OF CATALYST COMPOSITION ON DIVINYL YIELD IN THE CATALYTIC CONVERSION OF ETHANOL

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Abstract

This study investigates the synthesis of highly active and productive catalysts for the conversion of ethanol to divinyl, along with an assessment of their physicochemical properties. The catalysts were synthesized based on mixed oxides of magnesium and aluminum, as well as zirconium-containing zeolites. Through hydrothermal methods and the use of various molecular sieves, catalyst samples with high sorption capacities were obtained. Their physicochemical properties were characterized using atomic emission spectrometry, X-ray phase analysis, and low-temperature nitrogen adsorption techniques. The catalytic activity and deactivation behavior of the catalysts were evaluated via the hydrogenation of crotonaldehyde. The results demonstrated that zirconium-based catalysts exhibit high selectivity and stability for divinyl production. From the perspective of economic efficiency and technological feasibility, zirconium-containing catalysts are identified as the most optimal for the catalytic synthesis of divinyl from ethanol.

Keywords: Ethanol, divinyl, catalyst, zirconium, iron and zinc oxides, mechanism, reaction yield.

Introduction

1,3-Butadiene is one of the key products in petrochemical synthesis. It is used as a monomer for the production of synthetic rubber and various types of plastics [1]. Butadiene is primarily used in the manufacture of polybutadiene and styrene-butadiene rubbers, which are critical raw materials for car tires, protective mechanical coatings, and various plastic products. As of 2024, global production capacity for butadiene exceeded 20 million tons, with an annual growth rate of approximately 5% [2,3], indicating its high global demand and strategic role in the petrochemical industry.

Currently, the main production technology is the pyrolysis of naphtha to obtain ethylene, with butadiene produced as a by-product. However, the yield of butadiene in this process remains relatively low (about 4–5%) [3–6]. During naphtha pyrolysis, high-temperature cracking generates ethylene, propylene, and other olefins along with butadiene. Existing production capacities based on naphtha cracking are insufficient to meet the growing consumer demand for butadiene, leading to shortages and prompting the search for alternative synthesis technologies [3,7].

Meanwhile, bioethanol production has rapidly developed worldwide in recent years. In 2021, over 120 billion liters of bioethanol were produced, and it is expected to grow steadily at a rate of 10–12% annually over the next decade [8–10]. Producing butadiene from bioethanol is considered a promising and eco-friendly approach, reducing both energy consumption and environmental impact. Furthermore, this route is relatively economical and efficient.

However, many studies examining catalyst activity have been conducted under conditions not representative of industrial ethanol-to-butadiene processes. For instance, authors in [12–20] often used highly diluted ethanol in an inert gas stream and applied only minimal catalyst loads. While such setups allow detailed analysis of catalyst activity, chemical behavior, and kinetics, they do not accurately reflect the catalyst's performance under actual industrial operating conditions.

Among known catalyst systems, Zn–Zr, La–Zn–Zr, Cu–Zr, and Cu–Zn oxides supported on silica gel have shown great promise in terms of availability and cost [18–20]. Using these catalysts, butadiene yields of up to 52.5% have been reported [20]. Zirconium-based catalysts, in particular, demonstrate excellent thermal stability and acid-base properties, contributing to increased reaction productivity.

1,3-Butadiene is a valuable conjugated diene commonly obtained as a by-product of steam cracking of naphtha in industrial applications. It is mainly used as a monomer in synthetic rubber production, with the majority of its consumption directed toward the automotive industry.

The most extensively studied catalyst for one-step ethanol-to-butadiene conversion is MgO/SiO_2, used in the industrial Lebedev process. To obtain highly selective and stable one-step catalysts, extensive research has been carried out involving modifications such as the addition of intermediate metals, replacement of MgO with one or more transition metal oxides, and changes in support materials (e.g., zeolites).

Materials and methods

Synthesis of Mixed Oxide Catalysts

Mixed oxides of magnesium and aluminum were synthesized via co-precipitation from corresponding salt solutions. Aqueous solution A containing 0.1 mol of $Mg(NO_3)_2 \cdot 6H_2O$ and 0.05 mol of $Al(NO_3)_3$ in 45 mL of water was slowly added to solution B, composed of 0.35 mol of NaOH and 0.09 mol of $Al(NO_3)_3 \cdot 9H_2O$ in 70 mL of water. The resulting suspension was stirred and heated at 333 K for 24 hours, then filtered and washed with distilled water. The obtained catalyst samples were dried at 393 K for 24 hours and subsequently calcined under a dry nitrogen flow at 723 K for 8 hours. Catalysts with various Mg/Al ratios were prepared by altering the concentration of Mg and Al nitrates in the starting solution to evaluate their catalytic efficiency for ethanol-to-divinyl conversion. Individual MgO and Al_2O_3 were prepared using similar procedures.

Hydrothermal Synthesis of Zr-Containing Materials

To synthesize mesoporous zeolite catalysts (Zr-MCZ) suitable for ethanol-to-divinyl conversion, a hydrothermal method was employed. First, 0.216 g of aluminum flakes were dissolved in 41.23 g of aqueous tetraethylammonium hydroxide (TEAOH), to which a suspension of 12 g of Aerosil in 29 g of water was added. The final gel composition was: 1.0 SiO_2: 0.56 TEAOH: 0.02 Al_2O_3: 15 H_2O. The gel was aged hydrothermally at 413 K for 72 hours. To remove aluminum, the resulting product was treated with 50 mL of 6 M nitric acid at 353 K for 24 hours. Following further hydrothermal aging at 413 K for 10 days, the synthesized samples were filtered, thoroughly washed with distilled water, and dried at 353 K. Template removal was carried out by calcination in an air stream at 823 K.

Physicochemical Characterization of Catalysts

Elemental Composition: The chemical composition of the catalyst samples was determined using two analytical techniques. Light elements such as Si and Mg were quantified via atomic emission spectroscopy. Prior to analysis, samples were digested using a mixture of HF and concentrated HNO_3, followed by dissolution in 1 M HCl. Tablet formation was conducted under 200 atm pressure for proper preparation of analytical samples. Measurement accuracy was within 5%.

Low-Temperature Nitrogen Adsorption: The porous structure of the catalyst samples was investigated using low-temperature nitrogen adsorption on an ASAP-2000 instrument at 77 K. Adsorption isotherms were recorded as V_adsorbed (cm^3/g) versus relative pressure (p/p_0). Micropore volumes were determined using the de Boer and Lippens t-plot method, and mesopore volume (10–200 Å) was estimated using the BJH (Barrett-Joyner-Halenda) method.

Transmission Electron Microscopy (TEM): TEM images were acquired at an accelerating voltage of 100 kV. Catalyst suspensions in ethanol were dropped onto a carbon-coated copper grid and dried at room temperature prior to imaging.

X-ray Diffraction (XRD) Analysis: Phase identification of synthesized catalysts was performed using a Phillips X'Pert diffractometer with CuKα radiation ($\lambda = 1.54051$ Å), scanning in the 2θ range of 5–50°. Crystalline phases were identified by comparing diffraction peaks to known phase reference patterns.

Thermogravimetric Analysis (TGA): The quantity and nature of coke deposits on spent catalysts were determined using thermogravimetric-differential thermal analysis on a SDT Q-600 (TA Instruments). Approximately 20–30 mg of catalyst was heated to 1023 K in dry air (flow rate: 100 mL/min) at a programmed heating rate of 10 °C/min to study oxidative decomposition.

Results and discussion

In the study of the reduction of crotonaldehyde to 1-hydroxy-2-butene, two methodological approaches were applied. The first approach involved the selective hydrogenation of crotonaldehyde using various high-activity and high-yield catalysts containing different metals, selected for their suitability for the conversion process. The second approach utilized the Meerwein–Ponndorf–Verley–Oppenauer (MPVO) reaction, where ethanol was employed as the hydrogen donor.

Catalytic Hydrogenation of Crotonaldehyde Using Metal-Based Catalysts

In the hydrogenation of crotonaldehyde to 1-hydroxy-2-butene, catalysts based on noble and transition metals from group IB of the periodic table, supported on silicon dioxide (SiO_2) and lanthanum oxide (La_2O_3), were investigated. These catalyst systems were evaluated for their high catalytic activity and selectivity in the target reaction.

The tested catalyst compositions, reaction conditions, and performance metrics are summarized in Table 1 (not shown here, assumed to follow this paragraph in your manuscript). These catalysts demonstrated varying levels of efficiency depending on the nature of the active metal component and the support material used.

The combination of precious metals with acidic and basic oxide supports facilitated the desired transformation under mild hydrogenation conditions. The choice of catalyst significantly influenced both the conversion rate of crotonaldehyde and the selectivity toward the desired unsaturated alcohol product.

Table 1. Properties of catalysts with high catalytic activity and productivity used for the hydrogenation of crotonaldehyde, selected for the process

A catalyst of choice for process implementation with high catalytic activity and performance	Metal content,%	Used salts
$5MgO*FeO*ZnO/SiO_2$	5.0	$Mg(NO_3)_2 \cdot 6H_2O$
$5La_2O_3*FeO*ZnO/SiO_2$	5.2	$La(NO_3)_3 \cdot 6H_2O$
$5ZrO_2*FeO*ZnO/SiO_2$	5.4	$ZrCl_4$
$5ZrO_2*FeO*ZnO/SiO_2$	5.0	$ZrO(NO_3)_2 \cdot 2H_2O$
$5Al_2O_3*FeO*ZnO/SiO_2$	5.2	$Al(NO_3)_3 \cdot 6H_2O$

All high-activity and high-yield catalysts selected for the process were prepared by incipient wetness impregnation using appropriate precursor solutions until the desired concentration was achieved. The metal contents determined by atomic emission spectroscopy corresponded well with the calculated values.

Main Products and Reaction Pathways

In all the studied catalysts with high catalytic activity and yield, crotonaldehyde underwent reduction to form a mixture of products. In addition to the target product, 1-hydroxy-2-butene, the formation of acetaldehyde, methyl ethyl ketone, butanal, and butanol was also observed.

- Acetaldehyde can be formed via the aldol condensation of crotonaldehyde in the presence of water.

- Methyl ethyl ketone (MEK) is generated through the isomerization of crotonaldehyde into methyl vinyl ketone, followed by hydrogenation of the C=C bond.
- Butanal is produced by non-selective hydrogenation at the C=C bond of crotonaldehyde.
- Butanol-1 is a fully hydrogenated product and may result from the reduction of either 1-hydroxy-2-butene or butanal.

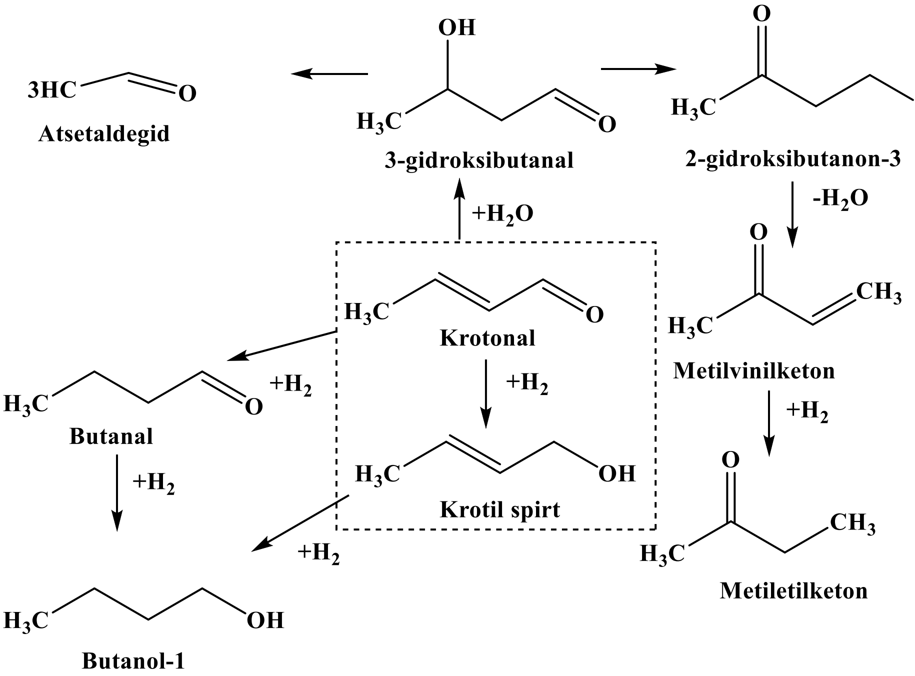

Figure 1. Scheme of the transformation of crotonaldehyde upon reduction with hydrogen.

Based on the presented data, it is evident that catalysts with high catalytic activity and yield, which are based on activated metals, do not provide a selectivity higher than 5% for the hydrogenation of crotonaldehyde. Moreover, all catalysts containing Mg, Zr, and La—selected for divinyl production from ethanol—show over 80% crotonaldehyde conversion. The primary hydrogenation product is butanal, and a significant amount of the fully hydrogenated product butanol-1 is also formed.

Thus, in catalysts incorporating metals with pronounced hydrogenating functions, the selective hydrogenation of crotonaldehyde does not achieve satisfactory selectivity due to non-selective hydrogenation of the C=C bond occurring in these metal systems.

In contrast, using the Meerwein–Ponndorf–Verley–Oppenauer (MPVO) reaction pathway for converting crotonaldehyde to 1-hydroxy-2-butene, catalysts based on zirconium oxide activated on SiO_2, ZrO_2, and La_2O_3, as well as zirconium-containing molecular sieves, were investigated. These catalysts were synthesized by co-precipitating the appropriate salts from solution, followed by filtration, drying, and calcination at 673 K.

The zirconium-based catalysts demonstrated type IV isotherms with a sharp uptake in the relative pressure region of $P/P_0 = 0.35$, corresponding to pores with an average diameter of ~30 Å. These activated catalysts had surface areas characteristic of amorphous materials, ranging from 80 to 270 m^2/g. The average pore diameter determined by the BJH method was approximately 100 Å for silica and 50 Å for lanthanum and zirconium oxides. Pure zirconium oxide exhibited a highly developed surface area (230 m²/g), comparable to that of $ZrO_2 \cdot FeO \cdot ZnO/SiO_2$.

Table 2. Characteristics of catalysts selected for the process of reducing crotonaldehyde with ethyl alcohol, with high catalytic activity and productivity

Catalyst samples	Chemical composition		Density area, m^2/g	Porosity volume, cm3/g	Micropore size, cm^3/g	Average pore diameter, Å
	rO_2	Si/Zr				
Zr-HSZs	1.52	134	470	0.29	0.20	7
Zr catalyst	1.07	184	870	1.1	-	30
ZrO_2*FeO*ZnO/SiO_2	1.90	105	270	0.70	-	100
ZrO_2/ZrO_2	1.85	-	80	0.08	-	50
ZrO_2/La_2O_3	2.02	-	80	0.08	-	50

Study of active centers of croton aldehyde reduction reaction. It is very desirable to have information about the nature and structure of the active centers in order to create a catalyst of choice for the process, which has high catalytic activity and productivity for the conversion of ethyl alcohol to divinyl. Calculations have shown that the reaction mechanism includes the following steps:

1) Adsorption of alcohol and ketone on Lewis acid centers;
2) deprotonation of alcohol to form alkoxide;
3) hydride transfer from the alkoxide to the carbonyl group of the ketone through a 6-membered transition state. Partially hydrolyzed Lewis acid centers have been proposed as active centers.

In this work, an attempt was made to find a correlation between the total number of acid centers detected by ammonia TPD and the catalytic activity and relative number of Lewis acids of various Zr-containing catalysts selected for the reduction of crotonaldehyde with ethyl alcohol, which had high catalytic activity and productivity. The surface OH groups were determined by IR spectroscopy of adsorbed CO. The results show that the catalytic activity does not depend on either the total number of acid centers or the number of surface OH groups. On the contrary, a good correlation was found with the number of Lewis acid centers of zirconium. The initial rate of formation of 1-hydroxy-2-butylene and the relative amount of Zr4+ Lewis acid centers increase in a series of catalysts selected for the process, which have the same high catalytic activity and yield: Zr-HSZs < ZrO_2*FeO*ZnO/SiO_2 < ZrO_2. The catalyst samples selected for the production of divinyl from ethyl alcohol, ZrO_2/ZrO_2 and ZrO_2/La_2O_3, which do not have Lewis Zr4+ centers, are completely inactive in the reaction. Thus, our results indicate that the Lewis centers of Zr4+ play a major role in the MPVO reaction. In order to determine the type of centers responsible for the formation of ethoxide particles, experiments were conducted to study the initial stages of the reaction on Zr-HSZs catalyst samples using IR spectroscopy. Deuteroethanol C_2H_5OD, dried with molecular sieve 3A, was used to distinguish the OH groups of the catalyst and substrate, which had high catalytic activity and productivity, selected for the process. C_2H_5OD was injected into the IR in small portions, and after equilibrium was established, the spectra of surface compounds were recorded.

When the mass fraction of acetaldehyde increases from 4 to 15%, no significant change in the selectivity for divinyl is observed, while the yield of divinyl increases from 9 to 28% (Fig. 2, Table 3). A further increase in the amount of acetaldehyde in the feedstock leads to a significant decrease in the selectivity for divinyl, and the selectivity for heavy adducts containing C6+ increases. This fact indicates that at high acetaldehyde concentrations, the yield of the aldol condensation increases, leading to a decrease in the selectivity of the entire process. The curve of the total conversion of acetaldehyde and ethyl alcohol passes through a maximum, which fits well with the reaction scheme, and the reduction of crotonaldehyde to 1-hydroxy-2-butylene requires a reducing agent, and the decrease in the content of ethyl alcohol leads to a decrease in the catalyst selected for the process, which has high catalytic activity and productivity.

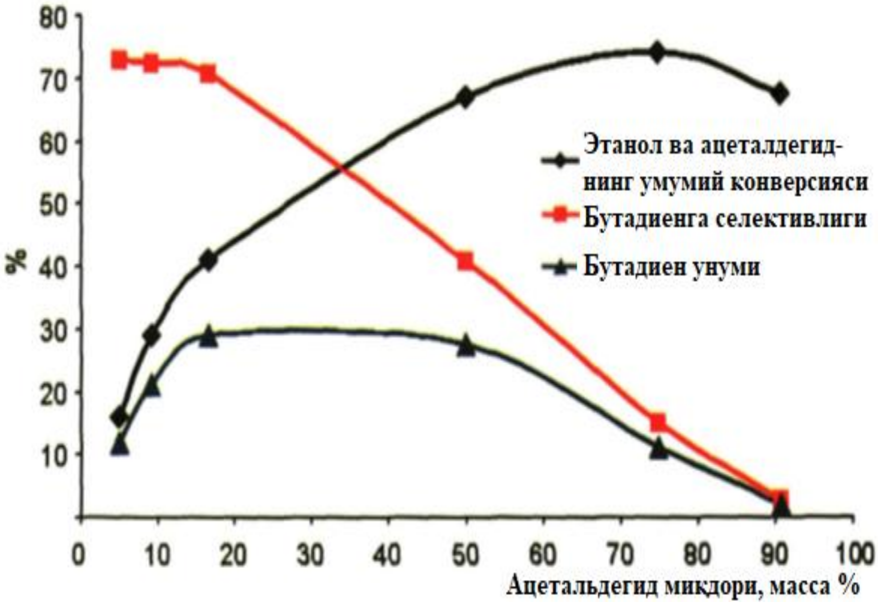

Figure 2. Dependence of raw material and selectivity of divinyl conversion on raw material composition (mass rate of raw material 0.3 g/g h, T = 593K)

Effect of type of metal. In the first stage, the effect of the methyl nature responsible for the hydrogenation of ethyl alcohol to acetaldehyde on the efficiency of the process was studied. For this purpose, catalyst samples selected for the production of divinyl from ethyl alcohol with high catalytic properties and productivity based on ZrO_2*FeO*ZnO/SiO_2 with various metals (Zr, La, Cu, Ni) were prepared and tested in the synthesis reaction of divinyl from ethyl alcohol. The results are presented in Table 3. The catalyst samples selected for the production of divinyl from ethyl alcohol with high catalytic properties and productivity based on copper and silver showed similar conversion and selectivity for divinyl (~ 74%). The catalyst selected for the process with high catalytic activity and productivity containing Zr has a slightly lower selectivity for divinyl (66%) due to the formation of butylenes.

Table 3. Effect of metal type in divinyl synthesis reaction (mass rate of raw material 0.3 g/g-h, T = 600K, reaction time 300 min)

	$0.3La/4ZrO_2*FeO*ZnO/SiO_2$	$0.3Ni/4ZrO_2*FeO*ZnO/SiO_2$	$0.3Cu/4ZrO_2*FeO*ZnO/SiO_2$
Conversion of ethyl alcohol to acetaldehyde,%	30.0	26.8	10.3
Product selectivity, %			
Divinyl	73.8	73.5	67.9
Ethylene	2.7	2.6	5.8
Propylene	3.0	3.7	3.7
Butylenes	2.9	3.6	5.7
Diethyl ether	3.7	3.3	6.4
Ethyl acetate	1.6	1.6	2.7
Butanol-1	3.0	3.4	2.6
Heavy products	9.3	8.4	5.3

To study the deactivation of catalysts with high catalytic activity and productivity, the dependence of the divinyl yield per unit mass of catalyst with high catalytic activity and productivity on the reaction time was analyzed (Fig. 3). The catalyst with high catalytic activity and productivity, containing Ni, showed good stability - the divinyl yield did not change almost during the 9-hour experiment. On the contrary, the catalysts with high catalytic activity and productivity, containing lanthanum and copper, were passivated. The fact that nickel can resist the deactivation of the catalyst with high catalytic activity and productivity, which is explained by the cleavage of coke precursors on the surface of the catalyst with high catalytic activity and productivity, is explained by the formation of coke precursors on the surface of the catalyst with high catalytic activity and productivity. Among the catalysts selected for the process, which have high catalytic activity and productivity, containing Cu and Ag, $0.3La/4ZrO_2/SiO_2$ showed high stability over time. It should also be noted that the selectivity towards the target product and by-products did not change during the experiment, which may indicate that the reaction mechanism does not change with the gradual poisoning of the active sites.

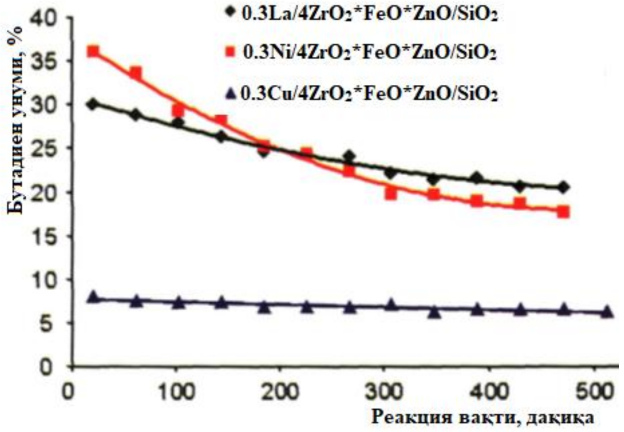

Figure 3. Dependence of divinyl yield on reaction time with various metals with catalysts having high catalytic activity and productivity, selected for the process (mass feed of raw material 0.3 g/g-h, T = 593 K).

Based on experimental data on the selectivity and stability of the target divinyl over time, a catalyst with high catalytic activity and productivity, including zirconium, was selected for further studies to carry out the process.

Effect of oxide type. In subsequent experiments, catalysts with high catalytic activity and productivity of various oxides were synthesized and studied. The catalyst with high catalytic activity and productivity of the selected catalyst samples for the production of divinyl from ethyl alcohol with high catalytic properties and productivity were compared at the same ethyl alcohol conversion, which was achieved by changing the mass flow rate of the feedstock. The catalytic activity of the catalyst with high catalytic activity and productivity of the selected catalyst for the process can be attributed to such a high concentration of acid centers, as well as the presence of Brønsted acid centers on the surface, which are active in the dehydration reaction. High yields of heavy products (up to 27%) were observed on the catalysts with high catalytic activity and productivity of ZrO_2 and MgO. This result indicates that magnesium and zirconium oxides are very active in the aldol condensation reaction, and it seems that the crotonaldehyde formed in the first step reacts with the subsequent acetaldehyde molecule to form unsaturated alcohols with carbonyl compounds at C6+. The reduction of the latter leads to the formation of dehydrating hexatrienes and their derivatives.

The highest selectivity for divinyl was demonstrated by the catalysts $0.3La/4ZrO_2{*}FeO{*}ZnO/SiO_2$ (74%) and 0.3La/Zr-HSZs (62%) with high catalytic activity and productivity, which were selected for the process, and which contain Zr-containing components responsible for the aldol condensation and reduction of crotonaldehyde. The catalyst Zr-HSZs with high catalytic activity and productivity, which was selected for the process, showed high resistance to deactivation during the first 4 hours of operation, but over time, the deactivation rate increased significantly due to the blockage of the pores of the mesoporous zeolite with high sorption and catalytic properties by compaction products. Other catalysts with high catalytic activity and productivity, selected for the process, had similar productivity over time. The maximum productivity of divinyl was observed at $0.3Zr/4ZrO_2{*}FeO{*}ZnO/SiO_2$.

Table 4. Effect of zirconium oxide content on the main parameters of divinyl synthesis reaction (mass consumption of raw materials 0.3 g/g-hour, T = 593K, reaction time 300 min)

	$Zr/4ZrO_2{*}FeO{*}ZnO/SiO_2$	$Zr/10ZrO_2{*}FeO{*}ZnO/SiO_2$	$Zr/18ZrO_2{*}FeO{*}ZnO/SiO_2$
Conversion of ethyl alcohol to acetaldehyde,%	48.0	45.6	45.8
Product selectivity, %			
Divinyl	72.1	73.0	72.8
Ethylene	1.5	2.1	2.3
Propylene	3.0	2.9	2.9
Butylenes	2.9	2.6	2.5
Diethyl ether	3.1	2.4	3.0
Ethyl acetate	3.0	2.9	3.0
Butanol-1	1.7	2.5	2.8
Heavy products	12.7	11.7	10.8

During the experiments, all the studied catalysts with high catalytic activity and productivity, selected for the process, were deactivated. At the same time, the deactivation rate changed slightly: with increasing ZrO_2 content, a slight decrease in the deactivation rate was observed, as indicated by a change in the slope of the divinyl productivity curve over time. This dependence indicates that the deactivation of the catalysts with high catalytic activity and productivity, selected for the process, may be associated with poisoning of the active centers of zirconium oxide.

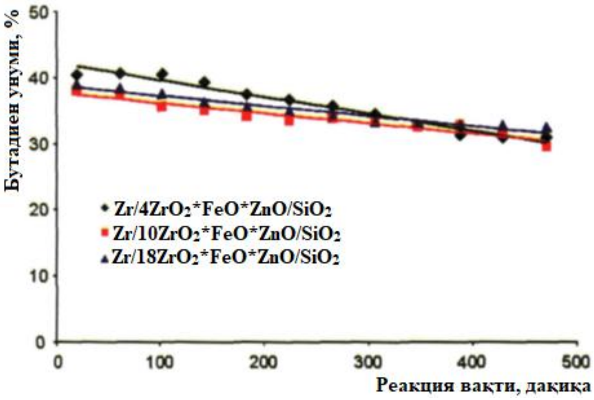

Figure 4. Dependence of divinyl yield on reaction time on catalysts with high catalytic activity and productivity containing zirconium oxide with different compositions (mass rate of raw material 0.3 g/g-h, T = 593 K).

Conclusions

Zirconium-containing catalysts with high catalytic activity and productivity in the process of divinyl production from ethyl alcohol show high selectivity and stability. These catalysts demonstrate high catalytic activity in the hydrogen reduction reaction of crotonaldehyde. Hydrothermal treatment during the synthesis process, the use of molecular sieves and X-ray phase analysis of the catalysts provide their high sorption properties. Experimental results have shown that the Lewis acid centers of zirconium are the main factor that plays an important role in the efficiency of the reaction. It is recommended to study the ways of modifying zirconium-containing catalysts and their long-term stability in future studies.

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