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FURFURAL HYDRATING ON PROMOTED SKELETAL COPPER CATALYSTS

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Abstract. *Introduction.* Furfural is a five-membered heterocyclic aldehyde of the furan series, which is an important biomass-derived compound. Its hydrogenation product, furfuryl alcohol, has numerous industrial applications. The selective hydrogenation of furfural has attracted interest for the development of efficient catalytic processes. In this study, attention is given to alloyed copper catalysts modified by ferroalloys for the hydrogenation process. *The purpose of this work* is to develop a process for the selective hydrogenation of furfural to furfuryl alcohol. *T*he researchers developed highly active suspended alloyed alumina-copper catalysts, incorporating ferroalloys as promoters. Specifically, skeletal copper catalysts with 70% aluminum content were prepared, and ferroalloy additives such as FMo (ferromolybdenum) and FMn (ferromanganese) were introduced. The catalytic activity was studied under different technological conditions by monitoring the hydrogenation of furfural. *Results and Discussion.* It was found that the promoting effect of FMo and FMn is associated with the formation of new $MeAl_3$ compounds, inclusions, and eutectoids in the alloys, which remain in the non-alkaline state of the leached catalysts. Among the tested multicomponent copper catalysts, the activity order increased as follows: Cu-70% Al < Cu-FMn < Cu-3% FMo < Cu-FMo + FMn. *Conclusion.* The study demonstrates that alloyed copper catalysts promoted with ferroalloys, particularly a combination of FMo and FMn, exhibit enhanced catalytic activity and selectivity in the hydrogenation of furfural to furfuryl alcohol. These findings contribute to the development of more efficient catalytic systems for biomass-derived chemical transformations.

Key words: hydrogenation, furfural, furfural alcohol, process, alloy, catalyst, promotion.

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1. Introduction

Carbohydrate-containing plant raw materials and its waste are quite promising alternative resource for the production of valuable chemical compounds. Their advantages are in renewability, or even practical inexhaustibility [1-5].

It is well known that climate change, the prospects for the development of oil resources and the rise in prices for them forced the world to look for alternative ways to acquire valuable chemical substances and fuels. In recent years, technologies for the production of the most important chemicals for the chemical and automotive industries from biomass are actively developed [6-8].

For example, furan derivatives are obtained by catalysis from sugars contained in biomass [6]. The furans are in the latest "technological roadmap" of biofuels for transport, compiled by the International Energy Agency [9].

The five-membered heterocyclic aldehyde of the furan series - furfural and products of its processing have long attracted the attention of researchers as an initial object for the synthesis of new compounds. [10].

Furfural is obtained by the catalytic dehydration of hexoses or pentoses [7].

Furfural is also present in bio-oil obtained as a result of rapid pyrolysis of biomass [11,12]. This brown viscous liquid contains many oxygen-containing compounds that give the oil undesirable properties, which makes it unsuitable as a fuel for transportation [13]. Hydrogenation of bio-oil eliminates the most reactive oxygen-containing groups, which, in turn, are the least desirable for all components [14-15].

Another of the most promising areas of chemical processing of furfural is its catalytic hydrogenation to furfuryl alcohol. This compound is in great demand in the chemical, textile, construction, engineering and aviation industries.

On the basis of furfuryl alcohol get anticorrosive materials, resistant to acids and alkalis. In a mixture with polyvinylbutinol, furyl resin is used as varnishes, mastics and adhesives. A mixture of furfuryl alcohol with nitric acid is used as a fuel for jet engines. Furfuryl alcohol based resins have a number of very valuable properties, including mechanical and thermal stability. In addition, furfuryl alcohol is the starting material for the synthesis of various furan and aliphatic products [16].

The authors [17] studied the hydrogenation of furfural, on a Cu / SiO_2 catalyst at 230-290 °C. Detailed kinetics, density function calculations (DFT) and spectroscopic studies were combined to research this reaction. It was established that the Langmuir-Hinshelwood model fits well with kinetic data and provides parameters of physical significance.

In this regard, the development of optimal catalysts for furfuryl is a significant problem. This is due to the fact that there is practically absent data of systematic research on the process hydrogenation of furfural on multicomponent copper catalysts. In particular, the influence of the nature of the metal of the catalyst, modifying additives, and technological parameters has not been studied.

2. Experimental part

The following multicomponent catalysts were the objects of the study: skeletal aluminum-copper (70% Al) catalysts with modifying additives of ferromolybdenum - FMO and ferrmanganese - FMn and simultaneously ferromolybdenum and ferromanganese;

The original alloys were prepared in a high-frequency melting furnace according to the previously developed technology. In the quartz crucible, the calculated amount of aluminum was loaded in the form of pieces of 3-5 mm in size and heated to a temperature of 1000–1100 °C.

Melt mixing was carried out with an induction field for 3-5 minutes. Then the melt was poured into graphite molds and cooled in air. In the future, the alloys were crushed by hand and sieved through a sieve with a cell diameter of 0.25 mm. The catalysts were prepared by leaching the alloys with a 20% aqueous solution of caustic soda in a boiling water bath for 1 hour. The main hydrogenated compound is the five-membered cyclic aldehyde of furan series – furfural.

Experiments on the hydrogenation of furfural on suspended catalysts were carried out in a special autoclave with a capacity of 250 cm^3 with vigorous stirring of the reaction medium. The washed catalyst together with an aqueous solution of furfural was loaded into the reactor.

Then the reactor was purged with hydrogen and sealed. The process of hydrogenation of furfural was controlled by sampling after 10 minutes. The duration of most experiments was 60 minutes. Reaction conditions varied over wide limits: temperature 40-120 °C, hydrogen pressure 4-12 MPa.

200 cm^3 of a 10% aqueous solution of freshly distilled furfural were used for hydrogenation. The amount of catalyst is 0.45 g. The catalyst activity was evaluated by the reaction rate (W) of hydrogenation, expressed in g of furfuryl alcohol (PS) per 1 g of catalyst per 1 hour (W, g / g·hour).

Analysis of the products of furfural hydrogenation was performed by gas-liquid chromatography on a Chromo-4 chromatograph with a flame ionization detector. Polyethylene glycol-6000 was used as the stationary phase, and the inert solid carrier was a chromatone with granule size of 0.10-0.25 mm.

The quantitative composition of the products was determined by peak area using correction factors.

3. Results and Discussion

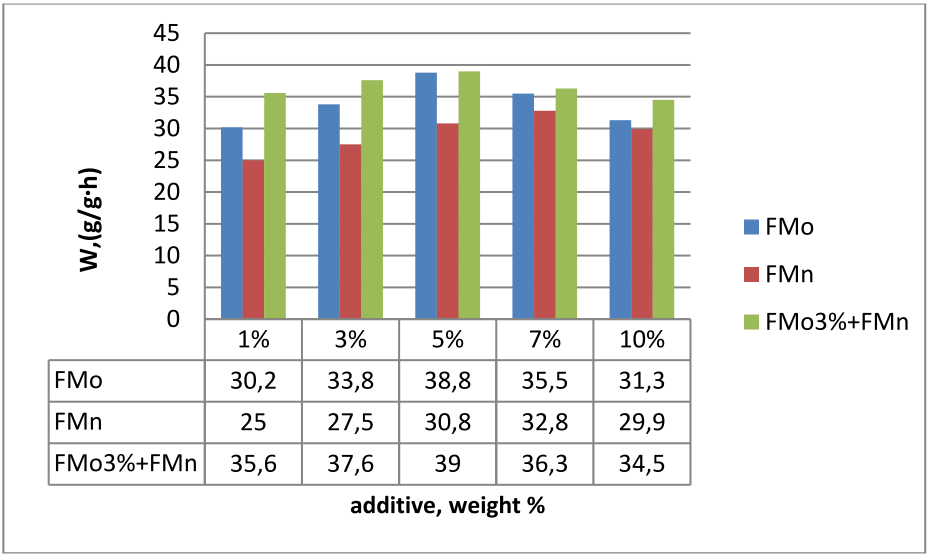
In this section, was studied the catalytic activity of skeletal copper (70% Al) catalysts with the additives of FMo, FMn, FMo, and FMn in the reaction of furfural hydrogenation under conditions of a wide variations of technological parameters. The results of the experiments are presented in table 1 and figure 1.

From the data table 1 it can be seen that hydrogenation of furfural on skeletal copper (70% Al) catalysts with the additives of ferroalloys is carried out selectively to ferroalloys, the rate of formation of which essentially depends on both the nature and the content of modifying ferroalloys.

Table 1 – The results of hydrogenation of furfural on skeletal copper (70% Al) catalysts with the additives of ferroalloys. Conditions: 200 cm^3 of a 10% aqueous solution of furfural; 6 MPa, 0.45 g of catalyst

Additive, weight %	t, ^0C	W, $\frac{g}{g \cdot h}$	K, h^{-1}	Ea, kDj/ mole
1	2	3	4	5
Cu(70%Al)	90 120	5.4 25.7	0.904 4.2751	58.9
1,0-10%FMo	60 90 120	27.3-29.4 34.1-35.3 42.0-44.6	11.2534-12.1310 16.6468-17.2530 24.4395-25.9346	14.0 18.4
1,0-10%FMn	60 90 120	21.4-27.7 28.3-33.8 38.4-45.0	3.5878-4.6126 4.7084-5.6311 6.4034-6.8411	25.8 28.9
+3%FMo +1-10% FMn	60 90 120	34.8-31.3 40.2-39.0 49.0-48.1	16.9775-17.5288 23.3882-22.6660 34.7815-34.0602	12.2 18.0

So, the addition of 1÷10% FMo or FMn, simultaneously 3% FMo and 1÷10% FMn in Cu-70% Al the alloy increase the rate of hydrogenation of furfural at 90 °C and 6 MPa, respectively, 6.3-8.1; 5.2-6.8 and 7.4-8.1 times. Cu-5% FMo, Cu-7% FMn, Cu-3% FMo-5% FMn catalysts are most active. The magnitude of the apparent ($E_{seeming}$) Activation energy of the hydrogenation process on promoted catalysts in the range of 60-900C ranges from 11.3-12.7 kJ / mol (on Cu-FMo catalysts), 20.9-22.8 kJ / mol (on Cu-FMn contacts) and 8.8-13.3 kJ / mol (on Cu-FMo-FMn catalysts), and in the range of 90-1200C - 13.4-16.0 kJ / mol (on Cu-FMo and Cu-3% FMo-FMn contacts), 25.0-26.0 kJ / mol (on Cu-FMn catalysts), and it mainly decreases with increasing activity of the catalysts. In general, modifying ferroalloys lower the activation energy of hydrogenation. In terms of $E_{seeming}$ it can be judged that the reaction is mainly limited by the activation of furfural on the surface. In fig. 1 graphic dependences of the rate of hydrogenation of furfural at 90 °C and 6 MPa on skeletal copper (70% Al) catalysts on the nature and content of ferroalloys in Cu-70% Al alloys are presented. From fig.1 it is seen that the curves of the rate of hydrogenation of furfural pass through the maxima corresponding to 5% FMo or 7% FMn, 3% FMo and 5% FMn.



	1%	3%	5%	7%	10%
FMo	30,2	33,8	38,8	35,5	31,3
FMn	25	27,5	30,8	32,8	29,9
FMo3%+FMn	35,6	37,6	39	36,3	34,5

Figure 1 – Dependence of the rate of hydrogenation of furfural at 900 °C and 6 MPa on the content of FMo (•), FMn (x), FMo and FMn (Δ) in copper alloys.

The promoting effect of FMo and FMn is due to the formation in alloys of new MeAl$_3$ compounds, inclusions and eutectoids, which non-alkaline states are part of leached catalysts. Modifying ferroalloys crush crystals of skeletal copper (70% Al), increase the specific surface, the concentration of the fraction of particles with $r_{max.} = 1$ μm, the pore volume and effective radii, the sorption capacity of catalysts for hydrogen.

They reduce the energy of the $K^{\sigma+}$–$H^{\sigma-}$ energy bond, which has a positive effect on the activity of skeletal copper (70% Al) in the reaction of selective hydrogenation of furfural to furfuryl alcohol.

The increase in the rate of hydrogenation from the content of alloying ferroalloys in the alloys is apparently due to the presence of their components in the alloy of the intermetallic compound CuAl$_2$, eutectic (Al + CuAl$_2$), non-leached compounds, inclusions.

The decrease in the activity of catalysts after maximum is associated with the formation of new compounds, inclusions, eutectoids, which are included in catalysts in undiluted states, increasing the particle size and thereby reducing the specific surface and the total number of active centers on the surface. The studied multicomponent copper (70% Al) catalysts with the addition of ferroalloys, according to their increasing activity, are arranged in a row: Cu-70% Al <Cu-FMn <Cu-3% FMo <Cu-FMo + FMn. This series approximately corresponds to an increase in the sorption capacity for hydrogen and the specific surface of the studied catalysts.

4. Conclusion

Thus, we have identified a certain dependence of the activity of the catalysts developed by us on their physicochemical properties. Comparison of the selectivity with data of granulometric composition and parameters of the porous structure shows that catalysts with an average particle diameter of 0.3-0.4 μm and a pore volume of 0.04-0.05 cm^3 / g with an effective radius of 28-31Å turned up most favorable for saturation > C = O – bond conjugated with a furan ring.

It was determined that the selectivity and stability of the catalysts depend on the binding energy of the adsorbed hydrogen. A significant weakening of the strengths and an increase in the volume of weakly adsorbed H_2 in promoted catalysts causes a violation of their (PS) selectivity. Modifying additives, which are included in the catalysts in the form of oxides, undiluted intermetallides, enrich the leaching products of relatively strongly adsorbed forms of H_2, which ensures high stability of contacts during long-term operation.

The activity and stability of the catalysts increase in the following sequence: Cu-70% Al <Cu-FMn <Cu-3% FMo <Cu-FMo + FMn. The selectivity of the process in this series increases in the reverse order.

Conflict of interests: The authors declare that there is no conflict of interest between the authors that requires disclosure in this article.

ПРОМОТРИЛЕНГЕН ҚАҢҚАЛЫҚ МЫС КАТАЛИЗАТОРЛАРЫНДА ФУРФУРОЛДЫҢ СУТЕКТЕНДІРУІ

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Түйіндеме: *Кіріспе.* Фурфурол — фуран қатарының бес мүшелі гетероциклді альдегиді, ол биомассадан алынатын маңызды қосылыс болып табылады. Оның гидрлену өнімі — фурфурил спирті — көптеген өнеркәсіптік салаларда қолданыс табады. Фурфуролды селективті гидрлеу тиімді каталитикалық процестерді жасауға үлкен қызығушылық тудырып отыр. Бұл зерттеуде фурфуролды гидрлеу процесінде ферроқоспалармен модификацияланған қорытпалы мыс катализаторларына ерекше назар аударылды. *Жұмыстың мақсаты* фурфуролды фурфурил спиртіне селективті түрде гидрлеу процесін жасау. Ғалымдар ферроқоспалармен күшейтілген, белсенділігі жоғары, алюминий мен мыс қорытпасынан жасалған, суспензия түріндегі катализаторларды әзірледі. Атап айтқанда, құрамында 70% алюминий бар қаңқалық мыс катализаторлары дайындалып, оларға ферромолибден (FMo) және ферромарганец (FMn) қосынды түрінде енгізілді. Каталитикалық белсенділік әртүрлі технологиялық жағдайларда фурфуролдың гидрленуін бақылау арқылы зерттелді. *Нәтижелер мен талқылау.* FMo мен FMn-ның күшейткіш әсері қорытпаларда жаңа $MeAl_3$ қосылыстарының, қосындылардың және эвтектоидтардың түзілуімен байланысты екені анықталды. Олар сілтісіз күйінде шайылған катализаторлардың құрамында сақталады. Зерттелген көп компонентті мыс катализаторлары арасында белсенділіктің өсу тәртібі келесідей болды: Cu-70% Al < Cu-FMn < Cu-3% FMo < Cu-FMo + FMn. *Қорытынды.* Зерттеу нәтижесінде ферроқоспалармен, әсіресе FMo мен FMn комбинациясымен күшейтілген қорытпалы мыс катализаторлары фурфуролды фурфурил спиртіне селективті түрде гидрлеу процесінде жоғары каталитикалық белсенділік пен селективтілік көрсететіні дәлелденді. Бұл нәтижелер биомассадан алынатын химиялық қосылыстардың тиімді түрленуін қамтамасыз ететін катализаторлық жүйелерді дамытуға өз үлесін қосады.

Түйінді сөздер: сутектендіру, фурфурол, фурфурилді спирт, процесс, құйма, катализатор, промотирлеу.

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ГИДРИРОВАНИЕ ФУРФУРОЛА НА ПРОМОТИРОВАННЫХ СКЕЛЕТНЫХ МЕДНЫХ КАТАЛИЗАТОРАХ

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Резюме. *Введение.* Фурфурол — это пятичленный гетероциклический альдегид фуранового ряда, представляющий собой важное соединение, получаемое из биомассы. Продукт его гидрирования — фурфуриловый спирт — широко используется в промышленности. Селективное гидрирование фурфурола вызывает интерес в контексте разработки эффективных каталитических процессов. В настоящем исследовании внимание уделено легированным медным катализаторам, модифицированным ферросплавами. *Цель и задачи.* Основная цель данной работы — разработать процесс селективного гидрирования фурфурола до фурфурилового спирта. *Методология.* Исследователи разработали высокоактивные суспензированные легированные катализаторы на основе оксида алюминия и меди с добавлением ферросплавов в качестве промоторов. Были приготовлены каркасные медные катализаторы с содержанием алюминия 70% и добавками ферромолибдена (FMo) и ферромарганца (FMn). Каталитическая активность исследовалась при различных технологических условиях путём мониторинга реакции гидрирования фурфурола. *Результаты и обсуждение.* Установлено, что усиливающее действие FMo и FMn связано с образованием новых соединений $MeAl_3$, включений и эвтектоидов в сплавах, которые сохраняются в некалийной форме в выщелоченных катализаторах. Среди исследованных многокомпонентных медных катализаторов активность возрастала в следующем порядке: Cu-70% Al < Cu-FMn < Cu-3% FMo < Cu-FMo + FMn. *Заключение.* Исследование показало, что легированные медные катализаторы, активированные ферросплавами, особенно комбинацией FMo и FMn, демонстрируют повышенную каталитическую активность и селективность в реакции гидрирования фурфурола до фурфурилового спирта. Эти результаты способствуют разработке более эффективных каталитических систем для превращения соединений, получаемых из биомассы.

Ключевые слова: гидрогенизация, фурфурол, фурфуриловый спирт, процесс, сплав, катализатор, промотирование.

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