ISSN 2518-1483 (Online),
ISSN 2224-5227 (Print)

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

№3
2025

ISSN 2518-1483 (Online),
ISSN 2224-5227 (Print)

2025 • 3

CENTRAL ASIAN ACADEMIC
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ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES

PUBLISHED SINCE JANUARY 1944

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ACADEMIC JOURNALOF PHYSICAL AND CHEMICAL SCIENCES

2025 • 3

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES.
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)

Owner: «Central Asian Academic Research Center» LLP (Almaty).

The certificate of registration of a periodical printed publication in the Committee of Information of the Ministry of Information and Social Development of the Republic of Kazakhstan **№ KZ93VPY00121157** issued **05.06.2025**

Thematic scope: *physics and chemistry.*

Periodicity: 4 times a year.

http://reports-science.kz/index.php/en/archive

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)
Меншіктеуші: «Орталық Азия академиялық ғылыми орталығы» ЖШС (Алматы қ.).
Ақпарат агенттігінің мерзімді баспасөз басылымын, ақпарат агенттігін және желілік басылымды қайта есепке қою туралы ҚР Мәдениет және Ақпарат министрлігі «Ақпарат комитеті» Республикалық мемлекеттік мекемесі **05.06.2025 ж.** берген **№ KZ93VPY00121157** Куәлік.
Тақырыптық бағыты: *физика, химия.*
Мерзімділігі: жылына 4 рет.
http://reports-science.kz/index.php/en/archive

ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)
Собственник: ТОО «Центрально-азиатский академический научный центр» (г. Алматы).
Свидетельство **№ KZ93VPY00121157** о повторной регистрации периодического печатного издания информационного агентства, информационного агентства и сетевого издания, выданное Республиканским государственным учреждением «Комитет информации» Министерства культуры и информации Республики Казахстан **05.06.2025**
Тематическая направленность: *физика, химия.*
Периодичность: 4 раза в год.
http://reports-science.kz/index.php/en/archive

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ACADEMIC JOURNAL OF PHYSICAL AND CHEMICAL SCIENCES
ISSN 2224-5227
Volume 3.
Number 355 (2025), 207–217

https://doi.org/10.32014/2025.2518-1483.374

UDC 544.478; 542.97; 546.264-31
IRSTI 31.15.28

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DRY REFORMING OF METHANE OVER THE HIGH ACTIVE Co-Fe-Ir-CONTAINING ALUMINA SUPPORTED CATALYST

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Abstract. Dry reforming of methane (DRM) is an efficient method for utilizing the main greenhouse gases – carbon dioxide and methane, where methane has even a stronger greenhouse impact than carbon dioxide. The reaction results in the formation of syngas – a mixture of hydrogen (H_2) and carbon monoxide (CO), which is an important building block for the chemical industry. Although this process is sufficiently well studied, searches for the effective catalysts continue. In this study, a new Co-Fe containing catalyst supported on alumina and modified with a small amount of Ir (0.2 mas.%) was synthesized, characterized by some physico-chemical studies such as TEM, SEM, BET, XRD, and H_2-TPR, and tested in dry reforming of methane. It was observed that the catalyst demonstrated high activity and stability in syngas production by carbon dioxide conversion of methane. The extents of conversion both of methane and carbon dioxide over the 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 were approximately the same ~ 95-96%, while syngas with a ratio of H_2:CO=1:1 was formed under the following reaction conditions: CH_4:CO_2=1:1, t=800°C, P=1 atm, GHSV – 1000 h^{-1}. No deactivation was observed for this catalyst over a period of 10 hours on stream. No sintering of highly

dispersed metal particles after reaction was shown by XRD and TEM. The catalyst with a composition of Co-Fe-Ir can be considered as a prospective way to design a highly stable and active catalyst for the CO_2-CH_4 conversion.

Keywords: Dry Reforming of Methane, Syngas, Supported Catalyst, Cobalt, Iron, Iridium

Funding: *This research was supported by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (grant # BR24992995).*

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АЛЮМИНИЙ ТОТЫҒЫНА ҚОНДЫРЫЛҒАН ЖОҒАРЫ БЕЛСЕНДІ Co-Fe-Ir ҚҰРАЙТЫН КАТАЛИЗАТОРДАҒЫ МЕТАННЫҢ ҚҰРҒАҚ РИФОРМИНГІ

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Аннотация. Метанның құрғақ риформингі (МҚР) негізгі парниктік газдарды – яғни көмірқышқыл газы мен метанды жоюдың тиімді әдісі болып табылады, және де метан көмірқышқыл газына қарағанда одан да күшті әсер етеді. Реакция химия өнеркәсібі үшін маңызды құрылыс материалы болып табылатын сутегі (H_2) мен көміртегі оксидінің (CO) қоспасы, синтез-газ түзілуіне әкеледі. Бұл процесс жақсы зерттелгенімен, тиімдірек катализаторларды іздеу әлі де жалғасуда. Бұл жұмыста бірқатар физикалық-химиялық әдістермен сипатталған трансмиссиялық электронды микроскопия (ТЕМ), сканерлеуші электронды микроскопия (СЭМ), БЭТ, рентгендік құрылымды талдау (XRD) және термиялық қалпына келтіру бағдарламасы (H_2-ТҚКБ), және метанның құрғақ риформинг процесінде сыналған алюминий оксидіне қондырылған және аз мөлшерде Ir (0,2 мас.%) мен

модификацияланған Co-Fe бар жаңа катализатор синтезделді. Катализатордың синтез-газ өндіруде жоғары белсенді және тұрақтылық танытатыны анықталды. Метан мен көмірқышқыл газының 10%Co-Fe-Ir(4,9-4,9-0,2)/Al_2O_3 катализаторындағы конверсия дәрежесі шамамен бірдей болып, 95–96% құрады, бұл реакция келесі жағдайларда жүргізіліп: CH_4:CO_2=1:1, t=800°C, P=1 атм, GHSV–1000 сағ$^{-1}$ сутегі мен көміртек тотығынан тұратын H_2:CO=1:1 қатынасында синтез-газ түзді. Бұл катализатордың 10 сағаттық жұмыс істеу барысында қатерсіздендіруі байқалмады. Рентгендік құрылымды талдау және трансмиссиялық электронды микроскопия әдістерімен жоғары дисперсті металл бөлшектерінің жентектелуі табылған жоқ. Co-Fe-Ir құрамды катализаторды CO_2-CH_4 парниктік газдарды түрлендіру үшін жоғары тұрақты және белсенді катализаторды әзірлеудің перспективті нұсқасы ретінде қарастыруға болады.

Түйін сөздер: метанның құрғақ риформингі, синтез-газ, қондырылған катализатор, кобальт, темір, иридий

Қаржыландыру: *Бұл жұмыс Қазақстан Республикасы Ғылым және жоғары білім министрлігі Ғылым комитетінің қаржылық қолдауымен орындалды (грант No BR24992995).*

© М.А. Жұмаш[1], К.Т. Тілеген[1,2], Е.А. Болеубаев[1], Ш.С. Иткулова[1,2*], 2025.
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СУХОЙ РИФОРМИНГ МЕТАНА НА ВЫСОКОАКТИВНОМ Co-Fe-Ir СОДЕРЖАЩЕМ НАНЕСЕННОМ НА ОКСИД АЛЮМИНИЯ КАТАЛИЗАТОРЕ

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Аннотация. Сухой риформинг метана (УКМ) является эффективным методом утилизации основных парниковых газов – диоксида углерода и метана, при

этом метан оказывает даже более сильное парниковое воздействие, чем диоксид углерода. Реакция приводит к образованию синтез-газа – смеси водорода (H_2) и оксида углерода (CO), который является важным строительным блоком для химической промышленности. Хотя этот процесс достаточно хорошо изучен, поиски более эффективных катализаторов до сих пор продолжаются. В данной работе был синтезирован новый катализатор, содержащий Co-Fe, нанесенный на оксид алюминия и модифицированный небольшим количеством Ir (0,2 мас.%), охарактеризованный рядом физико-химических методов, таких как просвечивающая электронная микроскопия (TEM), сканирующая электронная микроскопия (SEM), БЭТ, рентгеноструктурный анализ (XRD) и термопрограммированное восстановление (H_2-ТПВ), и испытанный в процессе сухого риформинга метана. Было обнаружено, что катализатор имеет высокую активность и стабильность в производстве синтез-газа. Степень превращения метана и диоксида углерода на катализаторе 10%Co-Fe-Ir(4,9-4,9-0,2)/Al_2O_3 была примерно одинаковой и составила 95-96%, при этом образовывался синтез-газ с соотношением H2:CO=1:1 при следующих условиях реакции: CH_4:CO_2=1:1, t=800 °C, P=1 атм, GHSV – 1000 $ч^{-1}$. Дезактивации этого катализатора за 10 часов работы не наблюдалось. Методами рентгеновской дифракции и просвечивающей электронной микроскопии не было обнаружено спекания высокодисперсных металлических частиц. Катализатор состава Co-Fe-Ir можно рассматривать как перспективный вариант для разработки высокостабильного и активного катализатора для конверсии парниковых газов CO_2-CH_4.

Ключевые слова: сухой риформинг метана, синтез-газ, нанесенный катализатор, кобальт, железо, иридий

Финансирование. *Работа выполнена при финансовой поддержке по программе целевого финансирования МОН РК ИРН BR24992995.*

Introduction. The increasing global energy demand due to population growth and the use of fossil fuels has led to environmental problems such as greenhouse gas emissions. This has ultimately led to many problems including climate change and global warming, which will affect people's living standards (Jeffry et al., 2021; Styring et al., 2023). Many strategies have been proposed to further reduce excessive greenhouse gas emissions (Su et al., 2022; Yang et al., 2025). Among the most promising methods to combat global warming, the process of the carbon dioxide conversion of methane, also called dry reforming of methane (DRM) has attracted considerable attention from both academia and industry. Carbon dioxide conversion of methane into syngas (a mixture of H_2 and CO) is a key initial reaction in a chemical technology production line that has dual functionality: a highly efficient method for producing intermediate fuel products and a method for capturing and neutralizing greenhouse gases (Eq.1) (Nguyen et al., 2024; Sharifnattaj et al., 2025). Dry reforming shows 74.8% lower net CO_2 emissions than industrially used steam reforming of methane (SMR). With the increasing carbon tax, dry reforming becomes the most cost-competitive route (Lee J. et al., 2025).

$$CH_4 + CO_2 \leftrightarrow 2CO + 2H_2 \ (\Delta H = +247 \text{ kJ/mol}) \qquad \text{(Eq.1)}$$

The main obstacle in the implementation of the DRM process is the high endothermicity of the reaction (Eq. 1) due to the exceptional stability of CH_4 and CO_2 molecules. To achieve the desired conversion degrees in the DRM process, elevated temperatures ranging from 700 to 1200°C are required (Olowoyo et al., 2024). DRM is a complex catalytic process with different reaction mechanisms depending on the catalyst and reaction conditions. The key step of the reforming process is believed to be the adsorption and decomposition of methane on the catalyst surface. At low temperatures, methane decomposition occurs gradually, and at high temperatures, it is completely decomposed to form carbon. Various experiments confirm that most of the carbon is formed through the methane decomposition reaction (Eq. 2). Carbon is easily deposited on the catalyst surface in the dry methane reforming reaction (Chen S. et al., 2020; Sun et al., 2024). This is the main cause of catalyst deactivation.

$$CH_4 \leftrightarrow C + 2H_2 \qquad \text{(Eq. 2)}$$

To realize the full potential of the DRM process, significant limitations need to be overcome, such as the strong endothermic properties and high activation energy of DRM, as well as coke formation. Suitable catalysts need to be developed to accelerate the reaction (Escalante et al., 2024). The most active and selective catalysts for DRM usually contain noble metals: Ir, Pt, Ru, Pd, and Rh, but their high cost makes them impractical for widespread use (Anil et al., 2020). However, high resistance to coke formation or the absence of carbonization of noble metals allows them to be used in small quantities as modifiers to accelerate the DRM (Boleubayev et al., 2023).

Materials and methods such as Co and Ni, with abundant reserves and low prices, exhibit excellent performance and are widely used as active metals in DRM. However, nickel has a strong tendency to form coke. At the same time, Co is characterized by sintering of metal particles, which is one of the main causes of its deactivation (Zafarnak S. et al., 2024). The coke resistance of catalysts depends on a number of factors, including the interaction between metals and between metal and support, the acid-base properties of the support, and the reducing and oxidizing properties of additives. Therefore, numerous studies are focused on the design of catalysts with high temperature stability and good thermal conductivity. Attention should be paid to controlling the particle size of the active metal during the preparation of the catalysts and their changes during the reaction.

The performance of catalysts used for the dry reforming of methane strongly depends on the selection of active metals, supports, and promoters. Support plays an important role in the adsorption and activation of carbon dioxide. Designing an economically viable catalyst that maintains high catalytic activity and stability can be achieved by exploiting the synergic effects of combining noble and/or non-noble metals to form highly active and stable bi- and tri-metallic catalysts (Aramouni et al., 2018; Yentekakis I., 2021). The secondary metals can modify the electronic and surface properties, thereby enhancing

catalytic performance and improving coke resistance. By fine-tuning the relationship between the structural characteristics and the catalytic activity of various metal centers, a pronounced synergistic effect can be achieved in a multifunctional catalyst system. This approach can significantly suppress coke formation on the surface and ensure prolonged catalytic stability (Ma et al., 2024).

The focus is now on more affordable materials as well as improving their properties through additives and new synthesis methods. Among metals, iron is less studied in DRM due to its lower activity. However, Fe catalysts have several advantages: they are resistant to coke formation at high temperatures, are cheaper than others, operate over a wide temperature range, and are well-suited for Fischer-Tropsch synthesis. Additionally, the redox properties of Fe species can contribute to improved reducibility, further enhancing the overall performance of the catalyst (Su et al., 2022).

In this research, the performance of Co-Fe-based catalysts with a mass ratio of Co: Fe = 1:1, modified with small amounts of iridium (0.2 mass%) and supported on alumina, was studied in DRM.

Experimental. A polymetallic catalyst containing Co-Fe-Ir was prepared by co-impregnation of alumina with the corresponding metal salts. The total amount of metals was 10 mas.%. The Co:Fe ratio was 1:1 and Ir was added to Co-Fe in an amount of 2 mas.%. That corresponds to the composition of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3. The synthesized catalyst was tested in the carbon dioxide conversion of methane (DRM). The DRM process was carried out in a quartz flow reactor under atmospheric pressure, a CH_4/CO_2 ratio was 1:1, a gas hourly space velocity (GHSV) was 1000 h^{-1}, and varying temperatures within a range of 400-800°C. The initial and final reaction products were online analyzed using gas chromatography (GC). The catalyst was characterized by using transmission electron microscopy (TEM), scanning electron microscopy (SEM), BET, X-ray diffraction (XRD), and H_2-TPR methods.

Results and discussion. The 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst was found to exhibit high activity in the CO_2 reforming of methane to produce synthesis gas. Figure 1 demonstrates the effect of temperature on the conversion of CH_4 and CO_2 (X_{CH4} and X_{CO2}, respectively) and the yield of H_2 and CO (Y_{H2} and Y_{CO}, respectively) under the following conditions: atmospheric pressure, CH_4-CO_2 ratio – 1:1, gas hourly space velocity (GHSV) – 1000 h^{-1}. In the range of 400-800 °C, methane and carbon dioxide conversion gradually rises from 1.5 to 95.9% and from 2.9 to 95.3%, respectively, and the yield of products increases from 0.15 to 13.37 and 0.66 to 13.28 micromoles per 1 gram of catalyst per second (μmol/g×s), respectively. In the range of 400-650°C, the conversion of CO_2 is slightly higher than the conversion of CH_4; however, a further increase in temperature to 700–800 °C leads to a nonsignificant predominance of CH_4 conversion.

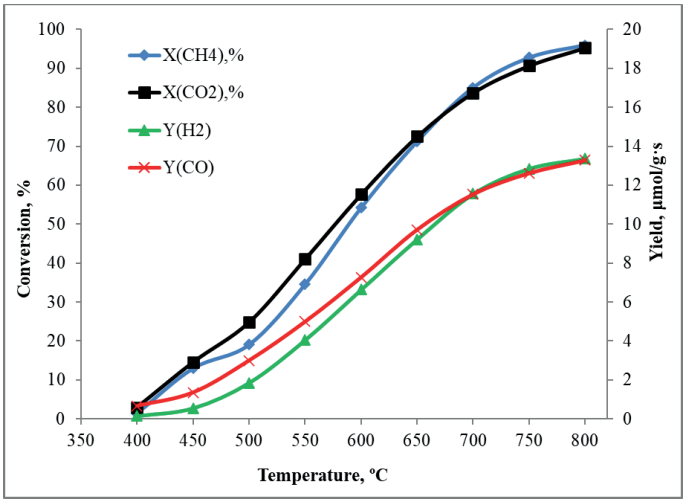

Figure 1 – Effect of temperature on CH_4 and CO_2 conversion and the yield of H_2, CO over 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst in DRM (CH_4:CO_2= 1:1, P = 0.1 MPa, GHSV = 1000 h^{-1})

A similar pattern is shown for the yields of H_2 and CO, which increase symbatically with temperature growth. Thus, at a temperature of 600°C, the yields of H_2 and CO are 6.64 and 7.27 µmol/(g×s), respectively. At 800°C, the yields of H_2 and CO are 13.37 and 13.28 µmol/(g×s), respectively. At lower temperatures, the resulting syngas has a H_2/CO ratio less than one, whereas with increasing temperature it exceeds one (Table 1).

Table 1 – DRM over 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 (CH_4:CO_2=1:1, P=0.1 MPa, GHSV=1000h^{-1})

t, °C	Conversion degree, %		Product yield, µmol/g×s		H_2/CO
	CH_4	CO_2	H_2	CO	
600	54.2	57.6	6.64	7.27	0.91
700	85.1	83.7	11.56	11.54	1.00
800	95.9	95.3	13.37	13.28	1.01

X-ray diffraction (XRD) analysis was performed for fresh (before reaction) and spent (after DRM) samples of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3. Both samples are X-ray amorphous. Reflexes attributed to the aluminum oxide phase (10-425, ASTM) are presented in both samples. In the fresh sample, the Co, Fe, and Ir phases are not detected (Fig. 2). The metallic Co phase appears after operation in DRM (ASTM 15-806). This can be caused by reductive effect of CO-H_2 medium, which is formed during the CO_2-CH_4 reaction. That indicates high-dispersed state of metals. TEM analysis confirmed a high-dispersed state of metals.

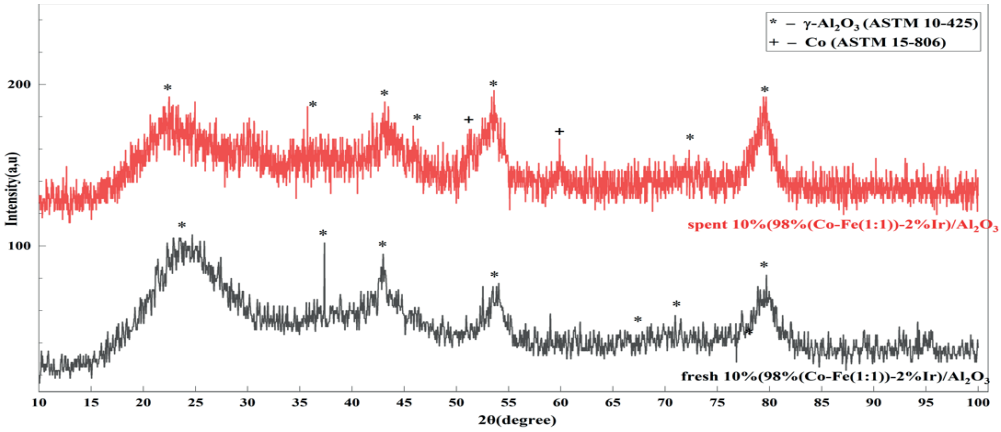

Figure 2 – XRD analysis of fresh and spent samples of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst

Elemental analysis and morphology of fresh and spent 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst samples were studied using SEM/EDS (scanning electron microscopy and energy-dispersive X-ray spectroscopy) (Fig.3). The chemical composition of the catalyst surface was determined using three spectra; the values were normalized and presented as mass percent. Average elemental composition values for fresh and spent samples is presented in Table 2.

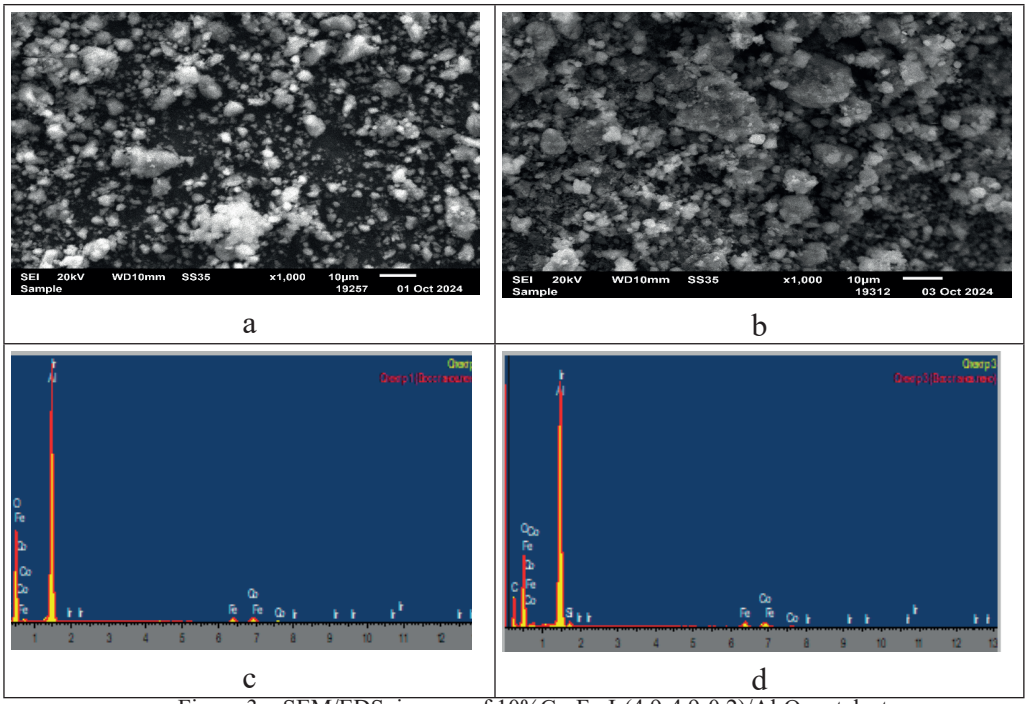

Figure 3 – SEM/EDS images of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst

Table 2 –EDS analysis data of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3

Sample	Composition, mas. %)						
	O	Al	Si	Fe	Co	Ir	Total
fresh	43.58	46.76	0	4.16	5.50	0.00	100
spent	39.12	49.54	0.48	4.80	6.06	0.00	100

Figure 3 shows the SEM images of the catalyst surface of the fresh and spent samples (3^a and 3^b, respectively) and results of elemental analysis of the fresh and spent samples (3^c and 3^d, respectively) of the catalyst. After operation of the 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst in DRM, an increase in iron and cobalt content was observed in the DRM, which may indicate the surface saturation with the active phase due to exposure to the reaction medium. This, in turn, causes a decrease in oxygen and aluminum content due to partial replacement of the oxide matrix by active components. Ir was not detected in the spectra of both fresh and spent samples, likely due to its low content. The appearance of silicon in the spent sample can be explained by contamination with quartz, which is used as an inert catalyst diluent to prevent localized overheating of the catalyst grains.

H_2-TPR analysis of the 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst demonstrate the presence of three peaks at 256, 275, and 376°C (Fig.4). They can be related with the stepwise reduction of iron (Jabbour et al., 2022) and cobalt (Nguen et al., 2004; Nurmakanov et al., 2016) oxides (Eqs. 3 and 4).

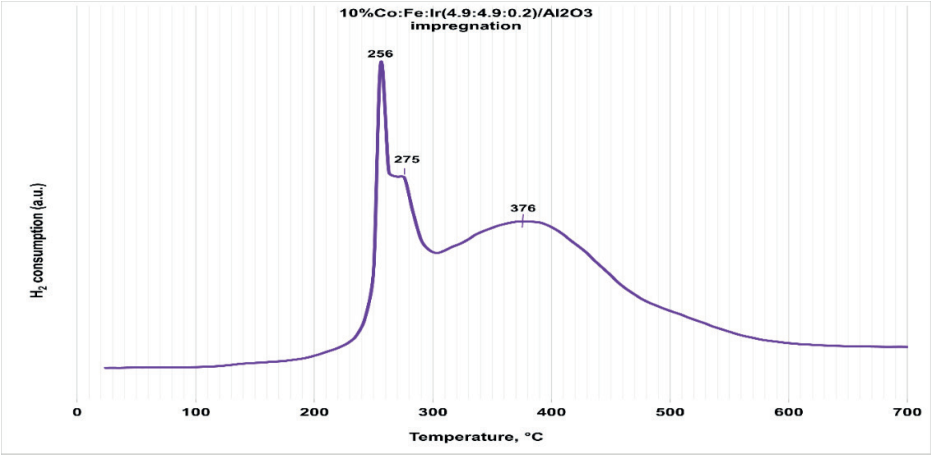

Figure 4 – H_2-TPR spectra of 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst

$$Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO \rightarrow Fe \quad \text{(Eq. 3)}$$

$$Co_3O_4 \rightarrow CoO \rightarrow Co \quad \text{(Eq.4)}$$

The highest temperature most likely corresponds to the reduction of iron oxide. Determining precisely which metal oxides are reduced at given temperatures is difficult due to possible peak overlap and the identical temperature regions for the reduction of

cobalt and iron oxides. It is known that incomplete reduction of $Fe_2O_{3(s)}$ iron oxide into metallic $Fe_{(s)}$, under hydrogen, is a challenge for iron-based catalysts limiting their industrialization for methane reforming or decomposition (Jabbour et al., 2022). The reduction temperatures of metal oxides in the studied polymetallic 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3 catalyst is quite lower than for individual metals. That is an evidence of synergetic effect because of Co-Fe-Ir interaction.

Conclusion. A new Co-Fe-Ir containing catalyst supported on alumina exhibits high activity in the process of carbon dioxide conversion of methane to synthesis gas. The degree of conversion of methane and carbon dioxide reaches ~ 95% at 800°C over the 10%Co-Fe-Ir(4.9-4.9-0.2)/Al_2O_3. Such particularly outstanding performance of Fe-Co catalysts modified with a small amount of Ir in DRM is due to their synergistic effects on activity and stability. Further studies of catalyst are planned in order to scale the synthesis gas production process using the DRM process.

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Ответственный редактор *А. Ботанқызы*
Редакторы: *Д.С. Аленов, Т. Апендиев*
Верстка на компьютере *Г.Д. Жадырановой*

Подписано в печать 3.09.2025.
Формат 60x88$^{1}/_{8}$.
18,0 п.л. Заказ 3.

«Central Asian Academic Research Center» LLP
Алматы, Қонаев к-сі, 142