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HYDROGENATION OF CARBON DIOXIDE ON Ni-Co/ZrO_2 CATALYSTS FOR CONVERSION INTO SYNTHETIC FUEL COMPONENTS

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Abstract. Catalytic hydrogenation of carbon dioxide is considered as a key process for the transition to a carbon-neutral economy, allowing the transformation of the main greenhouse gas into valuable chemical raw materials and components of synthetic fuels. The development of highly effective catalysts active under mild conditions is an urgent scientific and practical task. The aim of the work is the synthesis of composite catalysts based on nickel, cobalt and zirconium oxides, using the solution combustion method, for the selective hydrogenation of CO2 to methane and a comprehensive study of the catalytic properties. The activity of the catalysts obtained from the initial mixture of Ni(NO3)2 - Co(NO3)2 - ZrO(NO3)2·xH2O + urea of various compositions was studied in a flow reactor at temperatures ranging from 200 to 400° C. It was found that the optimal conditions for obtaining methane are: CO_2 conversion of more than 93,6%, CH_4 selectivity of 97%, $H_2 : CO_2 = 4 : 1$, T = 300°C, space velocity of 24,000

h^{-1}. The comprehensive characteristics of samples included X-ray phase analysis and scanning electron microscopy. X-ray phase analysis confirmed the formation of target oxide phases. High indicators are due to the synergistic effect between nickel, cobalt and zirconium oxides, as well as structural features formed due to the synthesis method. Research on Ni-Co/ZrO_2 catalysts for CO_2 hydrogenation is a timely and vital endeavor with clear environmental, economic, and strategic energy benefits. Innovation in this field is essential for turning the vision of a circular carbon economy into a practical reality.

Keywords: carbon dioxide, catalytic hydrogenation, Ni-Co/ZrO_2 catalyst, solution combustion method, methane

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КӨМІРҚЫШҚЫЛ ГАЗЫН СИНТЕТИКАЛЫҚ ОТЫН КОМПОНЕНТТЕРІНЕ АЙНАЛДЫРУ ҮШІН Ni-Co/ZrO_2 КАТАЛИЗАТОРЛАРЫНДА ГИДРЛЕУ

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Аннотация. Көмірқышқыл газының каталитикалық гидрленуі негізгі парниктік газды бағалы химиялық шикізатқа және синтетикалық отынның компоненттеріне айналдыруға мүмкіндік беретін көміртекті бейтарап экономикаға көшудің негізгі процесі болып саналады. Жеңіл жағдайларда белсенді, жоғары тиімді катализаторларды жасау өзекті ғылыми және практикалық міндет болып табылады. Жұмыстың мақсаты — көмірқышқыл газын метанға селективті гидрлеу үшін ерітіндіде жану әдісін қолдана отырып, никель, кобальт және цирконий оксидтері негізіндегі композитті катализаторларды синтездеу және каталитикалық қасиеттерін кешенді зерттеу. Әр түрлі пайыздық құрамды $Ni(NO_3)_2$ - $Co(NO_3)_2$ - $ZrO(NO_3)_2 \cdot xH_2O$ + мочевина бастапқы қоспасынан алынған катализаторлардың белсенділігі 200 -400° С температура диапазонында ағынды реакторда зерттелді. Метанды өндірудің оңтайлы шарттары: CO_2 конверсиясы 93,6% - дан жоғары, CH_4 селективтілігі 97%, $H_2 : CO_2 = 4 : 1$, T = 300°C, көлемдік жылдамдығы 24,000 $сағ^{-1}$ екені анықталды. Үлгілердің кешенді сипаттамасы рентгендік фазалық талдауды, сканерлеуші электронды микроскопияны қамтиды. Рентгендік фазалық талдау мақсатты оксид фазаларының түзілуін растады. Алынған жоғары мәндер никель, кобальт және цирконий оксидтері арасындағы синергиялық әсерге, сондай-ақ синтез әдісімен түзілген құрылымдық ерекшеліктерге байланысты. Көмірқышқыл газын гидрлеуге арналған, ерітіндіде жану әдісімен дайындалған $Ni\text{-}Co/ZrO_2$ композитті катализаторларын зерттеу экологиялық, экономикалық және стратегиялық энергетикалық пайдасы бар өзекті және маңызды жұмыс болып табылады. Осы саладағы инновациялар айналмалы көміртекті экономика тұжырымдамасын практикалық шындыққа айналдыру үшін аса қажет болып табылады.

Түйін сөздер: көмірқышқыл газы, каталитикалық гидрлеу, $Ni\text{-}Co/ZrO_2$ катализаторы, ерітіндіде жану әдісі, метан

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ГИДРИРОВАНИЕ ДИОКСИДА УГЛЕРОДА НА $Ni\text{-}Co/ZrO_2$ КАТАЛИЗАТОРАХ ДЛЯ ПРЕОБРАЗОВАНИЯ В КОМПОНЕНТЫ СИНТЕТИЧЕСКОГО ТОПЛИВА

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Аннотация. Каталитическое гидрирование диоксида углерода рассматривается как ключевой процесс для перехода к углеродно-нейтральной экономике, позволяющий трансформировать основной парниковый газ в ценное химическое сырьё и компоненты синтетических топлив. Разработка высокоэффективных катализаторов, активных в мягких условиях, является актуальной научно-практической задачей. Целью работы является синтез композитных катализаторов на основе оксидов никеля, кобальта и циркония с использованием метода растворного горения для селективного гидрирования диоксида углерода до метана и всестороннее изучение их каталитических свойств. Активность катализаторов, полученных из исходной смеси $Ni(NO_3)_2$, $Co(NO_3)_2$ и $ZrO(NO_3)_2 \cdot xH_2O$ с добавлением мочевины различного процентного состава, была исследована в проточном реакторе при температурном диапазоне 200–400 °C. Было установлено, что оптимальными условиями получения метана являются: конверсия CO_2 более 93,6%, селективность CH_4 — 97%, мольное соотношение $H_2 : CO_2 = 4 : 1$, температура 300 °C, объемная скорость 24 000 $ч^{-1}$. Комплексная характеристика полученных образцов включала рентгенофазовый анализ и сканирующую электронную микроскопию. Рентгенофазовый анализ подтвердил формирование целевых оксидных фаз. Высокие каталитические показатели обусловлены синергетическим эффектом между оксидами никеля, кобальта и циркония, а также особенностями структуры, сформированными вследствие выбранного метода синтеза. Исследование композитных катализаторов $Ni–Co/ZrO_2$, приготовленных методом растворного горения, для гидрирования диоксида углерода является актуальным направлением, обладающим выраженными экологическими, экономическими и стратегическими энергетическими преимуществами. Дальнейшие инновации в этой области необходимы для продвижения концепции циклической углеродной экономики к практической реализации.

Ключевые слова: диоксид углерода, каталитическое гидрирование, катализатор $Ni-Co/ZrO_2$, метод растворного горения, метан

Introduction. The anthropogenic emissions of carbon dioxide from fossil fuel combustion have reached unprecedented levels, driving global climate change and environmental instability. With atmospheric CO_2 concentrations exceeding 410 ppm

and continuing to rise at 3-4 ppm annually, there is an urgent need for effective strategies to mitigate carbon emissions while meeting growing global energy demands (Hasan, et al., 2020). The utilization of CO_2 as a chemical feedstock represents a promising approach to addressing this dual challenge, transforming a waste product into valuable fuels and chemicals through catalytic hydrogenation processes. This strategy not only helps reduce atmospheric CO_2 levels but also contributes to a more sustainable energy economy by providing alternative pathways for fuel production (Wei et al., 2021; Cui et al., 2024).

The hydrogenation of CO_2 to synthetic fuels has gained significant attention as a potential component of the broader carbon capture and utilization (CCU) paradigm. Unlike carbon capture and storage, which focuses solely on sequestration, CCU aims to valorize CO_2 by converting it into useful products, thereby creating economic incentives for emission reduction. The concept of Power-to-Gas technology, which converts renewable electricity into gaseous energy carriers via hydrogen production and subsequent CO_2 methanation, has emerged as a particularly promising approach for energy storage and distribution (Khajonvittayakul et al., 2021). This process enables the storage of intermittent renewable energy in the form of chemical bonds, providing a solution to the challenge of grid-scale energy storage while simultaneously utilizing captured CO_2.

Nickel-based catalysts have emerged as the most widely studied non-noble metal systems for CO_2 hydrogenation due to their high activity, relatively low cost, and availability. However, monometallic Ni catalysts suffer from several limitations, including sintering at elevated temperatures, coking (carbon deposition), and deactivation under reaction conditions (Cui et al., 2024). The addition of cobalt as a promoter has been shown to enhance catalytic performance through several mechanisms: improving reducibility, modifying surface properties, and increasing resistance to deactivation. The combination of Ni and Co creates a bimetallic synergy that often results in superior performance compared to either metal alone (Hasan et al., 2020).

The choice of support material plays an equally crucial role in determining catalyst performance. Zirconium dioxide has proven to be an excellent support for CO_2 hydrogenation catalysts due to its unique combination of redox properties, surface acidity/basicity, and oxygen storage capacity. Unlike conventional supports such as Al_2O_3, ZrO_2 exhibits weak hydrophilic character, which reduces the poisoning effect of water – a major byproduct of methanation reactions – on active sites (Phongamwong et al., 2017; Wang et al., 2019). Additionally, ZrO_2 can enhance metal dispersion and create strong metal-support interactions that stabilize active particles against sintering. The presence of ZrO_2 in the catalyst formulation helps suppress the reverse water gas shift reaction by providing basic sites that favor CO_2 adsorption in a configuration conducive to direct hydrogenation rather than CO formation (Wang et al., 2019).

The specific reaction intermediates and their stability on the catalyst surface play a crucial role in determining the overall activity and selectivity. For instance, the presence of oxygen vacancies on the ZrO_2 surface facilitates the activation of CO_2 molecules, while the combination of Ni and Co sites optimizes the dissociation of H_2 and the

subsequent hydrogenation steps (El-Salamony et al., 2023). Understanding these mechanistic details is essential for the rational design of more efficient catalysts tailored for specific product distributions.

The solution combustion method was chosen as a method for preparing the catalyst due to its significant advantages: simplicity, speed, low temperature and energy efficiency. This method allows obtaining nanosized powders with a high degree of homogeneity, developed surface and a given composition, which is extremely important for creating highly efficient catalytic systems.

The SCS process promotes the formation of strong metal-support interactions and synergistic effects between components, which are crucial for enhancing catalytic performance in CO_2 hydrogenation reactions. The intimate mixing of Ni, Co, and Zr precursors at the molecular level in the initial solution, combined with the rapid combustion process, facilitates the formation of interfaces between metallic nanoparticles and the ZrO_2 support, which often serve as active sites for CO_2 activation and hydrogenation (Summa et al., 2022; Zhao, et al., 2016).

Studies have shown that SCS-prepared catalysts typically exhibit higher specific activities (up to 2-3 times higher) compared to counterparts prepared by conventional methods such as impregnation or co-precipitation. For instance, Ni/ZrO_2 catalysts prepared via SCS using urea as fuel demonstrated CO_2 conversions exceeding 85% with methane selectivities above 95% at relatively mild temperatures (300 – 350°C), significantly outperforming impregnated catalysts which showed lower conversions (70 – 75%) and faster deactivation (Zhao et al., 2016). This paper addresses this gap by presenting activity data for a novel solution combustion synthesis (SCS)-derived catalyst based on a Ni-Co-Zr formulation, which demonstrates high efficacy for the catalytic hydrogenation of carbon dioxide into methane.

Materials and methods.

Catalyst preparation

A series of catalysts on the base of (10 % $Ni(NO_3)_2$ + 40 % $Co(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea, 20 % $Ni(NO_3)_2$ + 30 % $Co(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea, 30 % $Ni(NO_3)_2$ + 20 % $Co(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea, 40 % $Ni(NO_3)_2$ + 10 % $Co(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea, 45 % $Ni(NO_3)_2$ + 5 % $Co(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea, 50 % $Ni(NO_3)_2$ + 50 % $ZrO(NO_3)_2 \cdot xH_2O$ / 50 % urea) was prepared by solution combustion synthesis method. The catalyst preparation process was as follows: a solution of salts in distilled water within a quartz glass was heated to 80 - 100°C and subsequently subjected to heat treatment in a preheated muffle furnace at initiation temperature. The synthesis reaction is initiated by intense heat release, which ensures the rapid propagation of a combustion front and a sharp, spike-like increase in temperature. This process leads to the formation of structured catalysts within minutes, a key factor responsible for their high catalytic activity. The final appearance of the obtained sample is shown in Figure 1.

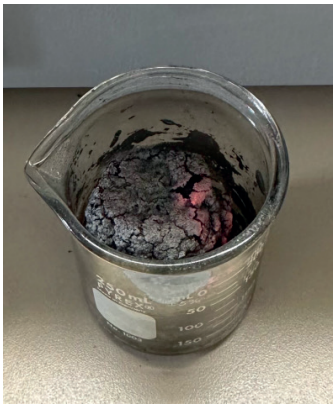

Figure 1 - Physical appearance of the SCS-derived catalyst

Characterization techniques

X-ray diffraction (XRD) analysis was performed using a DRON - 4 - 0.7 diffractometer equipped with a Co - Kα radiation source. Scans were recorded in the 2θ range of 6 - 100° with the following parameters: a step size of 0,020°, a count time of 0,6 s/step, and a goniometer speed of 2°/min. A maximum of 821 pulses were recorded. Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Spectroscopy (EDS) were conducted on a JEOL JSM - 6610LV instrument to examine the sample's morphology and elemental distribution. Prior to analysis, samples were sputter-coated with a 5 - 10 nm carbon layer. Mechanical properties were assessed by compressive strength tests performed on cylindrical specimens (Ø1 cm × 2 cm) using a 100 kN, strain-controlled universal tester at a constant displacement rate of 100 μm/min. Chemical analysis of the initial mixture and reaction products was carried out with a "Chromos GC - 1000" gas chromatograph. Chromatographic peaks were identified and calculated using the instrument's software, which utilized calibration curves constructed from pure substances.

Catalytic activity studies

The catalytic activity of the SCS-synthesized materials was evaluated in a fixed-bed flow reactor within an automated laboratory unit, equipped for online analysis of reactants and products. The reaction studied was the hydrogenation of CO_2 to synthetic fuel components, performed under atmospheric pressure using a feed gas mixture of H_2 : CO_2 : Ar (76 : 19 : 5 molar ratio). The catalyst was pre-reduced in situ prior to each catalytic test in a 11 % H_2/Ar mixture (100 ml/min) at 400°C for 1 h. The tests were conducted across a temperature range of 200 - 400°C and at space velocities of 12,000 - 48,000 h^{-1}. The reproducibility of the data was rigorously confirmed. To this end, measurements at each specific temperature were repeated in triplicate until fully consistent results were obtained. A prolonged stability assessment was conducted on the optimal catalyst (45% Ni + 5% Co + 50% Zr) for a time-on-stream (TOS) of 27 hours. The catalyst maintained its high catalytic activity with no observable deactivation, indicating excellent structural and chemical stability under the reaction conditions.

Results and discussions. Based on a review of literature data, optimal testing conditions for the Ni - Co - Zr catalysts were set at a gas hourly space velocity (GHSV) of 24,000 h^{-1} and a temperature range of 200 - 400°C. Repeatability testing determined that the peak yield for the conversion of carbon dioxide to methane was achieved at 300°C. The results presented in Table 1 indicate that the catalyst with a composition of 45 % Ni + 5 % Co + 50 % Zr, prepared with 50 % urea, demonstrated the highest activity, achieving a CH_4 yield of 99,2%.

Table 1 - Catalysts prepared by SCS method for the hydrogenation of carbon dioxide to methane

Chemical composition of catalysts, % wt.	Yield of CH_4, %	Selectivity of CH_4, %
10 % Ni + 40 % Co + 50 % Zr + 50 % urea	21,2	91,5
20 % Ni + 30 % Co + 50 % Zr + 50 % urea	40,4	95,5
30 % Ni + 20 % Co + 50 % Zr + 50 % urea	34	91,8
40 % Ni + 10 % Co + 50 % Zr + 50 % urea	93,7	97
45 % Ni + 5 % Co + 50 % Zr + 50 % urea	99,2	97,1
50 % Ni + 50 % Zr + 50 % urea	75,5	95,7

Figure 2 shows the conversion of CO_2 by 10% Ni + 40% Co + 50% Zr, 20% Ni + 30% Co + 50% Zr, 30% Ni + 20% Co + 50% Zr, 40% Ni + 10%Co + 50% Zr and 45% Ni + 5% Co + 50% Zr catalysts in the temperature range of 200-400°C.

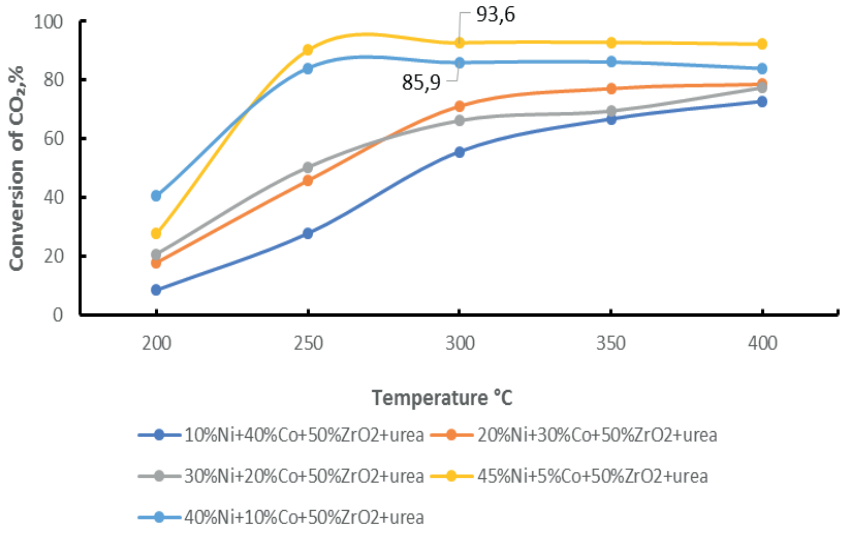

Figure 2 - Conversion of CO_2 over the 10% Ni + 40% Co + 50% Zr, 20% Ni + 30% Co + 50% Zr, 30% Ni + 20% Co + 50% Zr, 40% Ni + 10%Co + 50% Zr and 45% Ni + 5% Co + 50% Zr + urea catalysts at 200-400°C

40% Ni + 10%Co + 50% Zr and 45% Ni + 5% Co + 50% Zr + urea catalysts exhibited high activity, with CO_2 conversions reaching 85,9% and 93,6%, respectively. Figures 3 and 4 show the dependences of methane selectivity on the process temperatures of two more active catalysts.

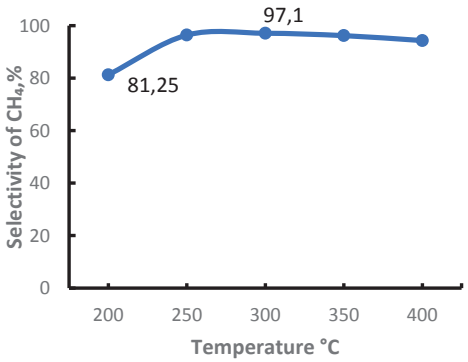

Figure 3 - Selectivity of CH_4 over the 45% Ni + 5% Co + 50% Zr + 50% urea catalyst at 200-400°C

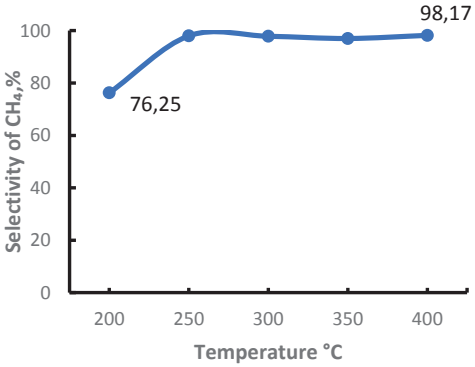

Figure 4 - Selectivity of CH_4 over the 40% Ni + 10% Co + 50% Zr + 50% urea catalyst at 200-400°C

The most active catalyst formulation (45 % Ni + 5 % Co + 50 % Zr, prepared with 50 % urea) was selected to study the effect of gas hourly space velocity on catalytic performance. As presented in Figure 5, CO_2 conversion exhibits an inverse relationship with GHSV.

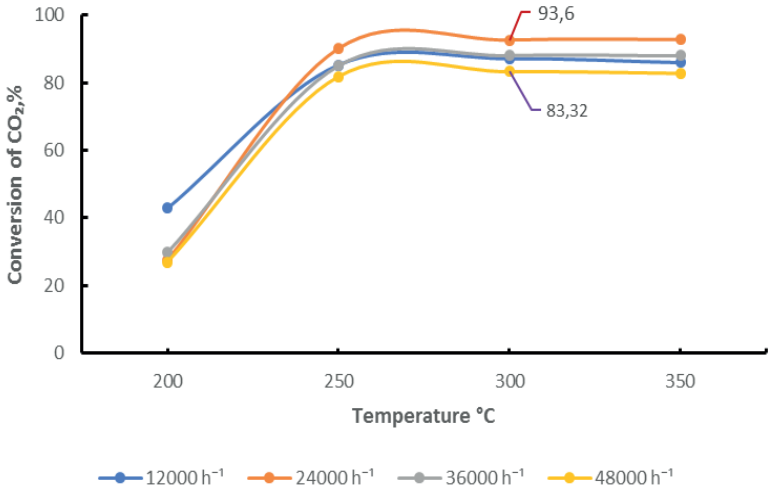

Figure 5 - Conversion of carbon dioxide over the 45 % Ni + 5 % Co + 50 % Zr + 50 % urea catalyst at different space velocities

This correlation is a classic manifestation of a mass transfer limitation, where elevated space velocities curtail the residence time, thereby insufficiently exposing the reactant molecules to the catalyst's active sites for complete conversion to methane.

The influence of reaction temperature on the methane (CH_4) production rate, expressed as space-time yield is shown in Figure 6. Data were obtained using a 45% Ni + 5% Co + 50% Zr + urea catalyst and a GHSV of 24,000 h^{-1}.

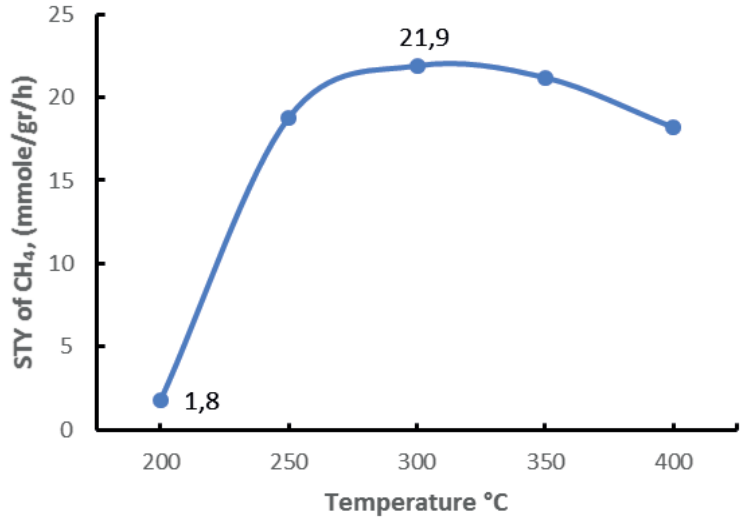

Figure 6 - Effect of the reaction temperature on the STY of CH_4, (mmole/gr/h) of process over 45%Ni + 5%Co + 50%Zr + urea catalyst at GHSV 24000 h^{-1}

This graph shows the space-time yield of methane in 200 and 300°C, respectively. Further, for comparison, the space-time yield of methane obtained from 40% Ni + 5% Co + 50% Zr + urea catalyst is shown in Figure 7.

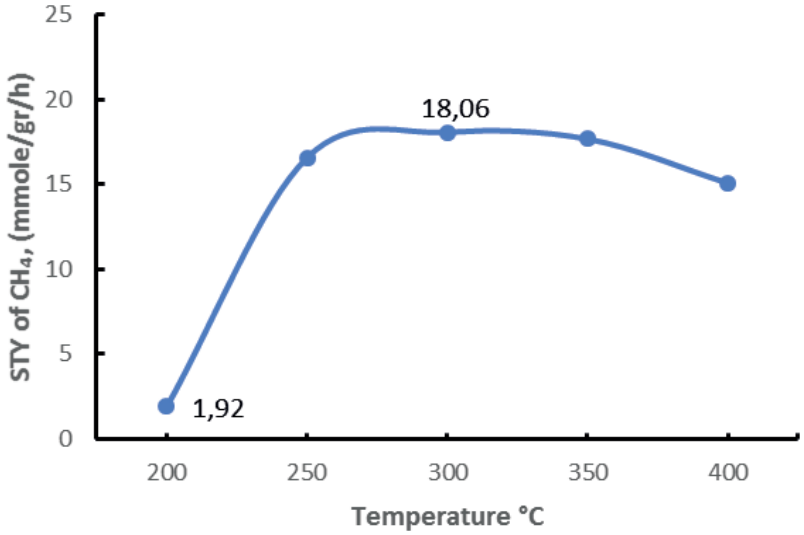

Figure 7 - Effect of the reaction temperature on the STY of CH_4, (mmole/gr/h) of process over 40%Ni + 5%Co + 50%Zr + urea catalyst at GHSV 24000 h^{-1}

Below, in Figures 8 and 9, the dependences of carbon dioxide conversion and methane selectivity to time on stream are illustrated, respectively.

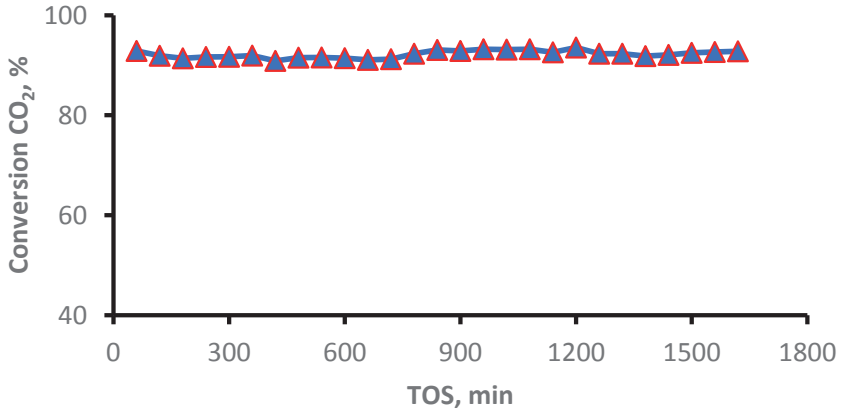

Figure 8 - Effect of the TOS on the conversion of CO_2

This graph shows that carbon dioxide conversion remained virtually unchanged over the 27 hours of the catalytic stability test.

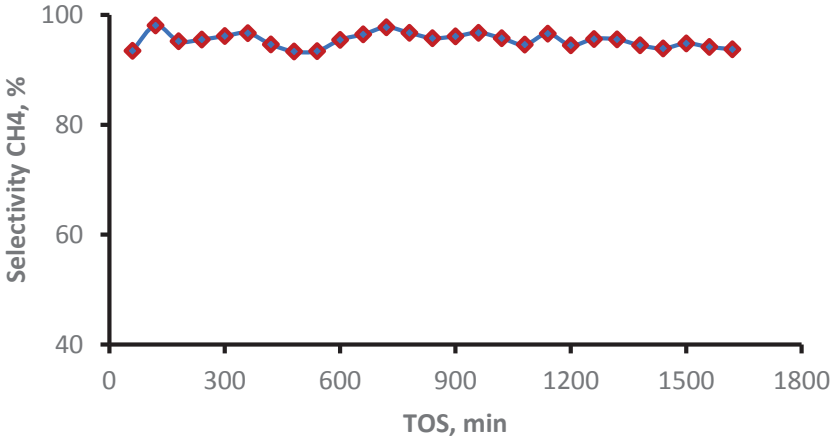

Figure 9 - Effect of the TOS on the selectivity CH_4

In this graph, it can be seen that the methane selectivity increased in places but did not drop below the initial value during the 27 hours of the catalytic stability test.

The phase composition of the catalyst was determined by X-ray diffraction. Figure 10 presents the XRD pattern of the 45 % Ni + 5 % Co + 50 % Zr + 50 % urea catalyst.

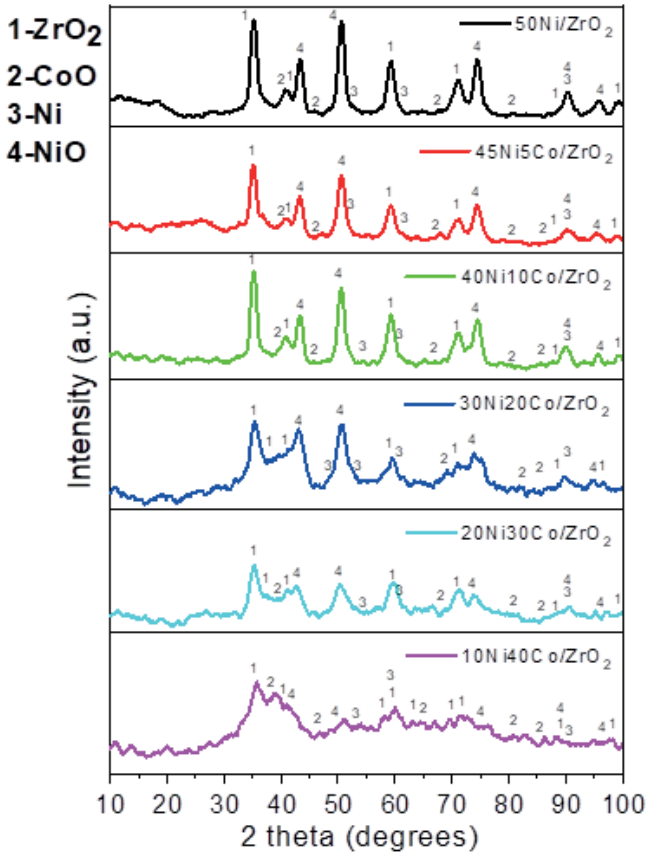

Figure 10 - XRD spectrum of the 45 % Ni + 5 % Co + 50 % Zr + 50 % urea catalyst

The analysis confirms the presence of metallic Ni, NiO, CoO, and monoclinic ZrO_2 phases. The identified diffraction peaks are consistent with their respective JCPDS standard reference patterns: metallic Ni (JCPDS 01-1260) with primary d-spacings at 2.039, 1.762, and 1.246 Å; NiO (JCPDS 44-1159) with d-spacings at 2.409, 2.088, 1.478, 1.476, 1.261, and 1.204 Å; CoO (JCPDS 42-1300) with d-spacings at 2.455, 2.135, 1.501, and 1.246 Å; ZrO_2 (JCPDS 17-0923) with d-spacings at 2.840, 2.610, 2.540, 1.832, 1.653, 1.602, 1.541, and 1.475 Å.

Scanning electron micrographs of all fresh catalyst samples are provided in Figure 11.

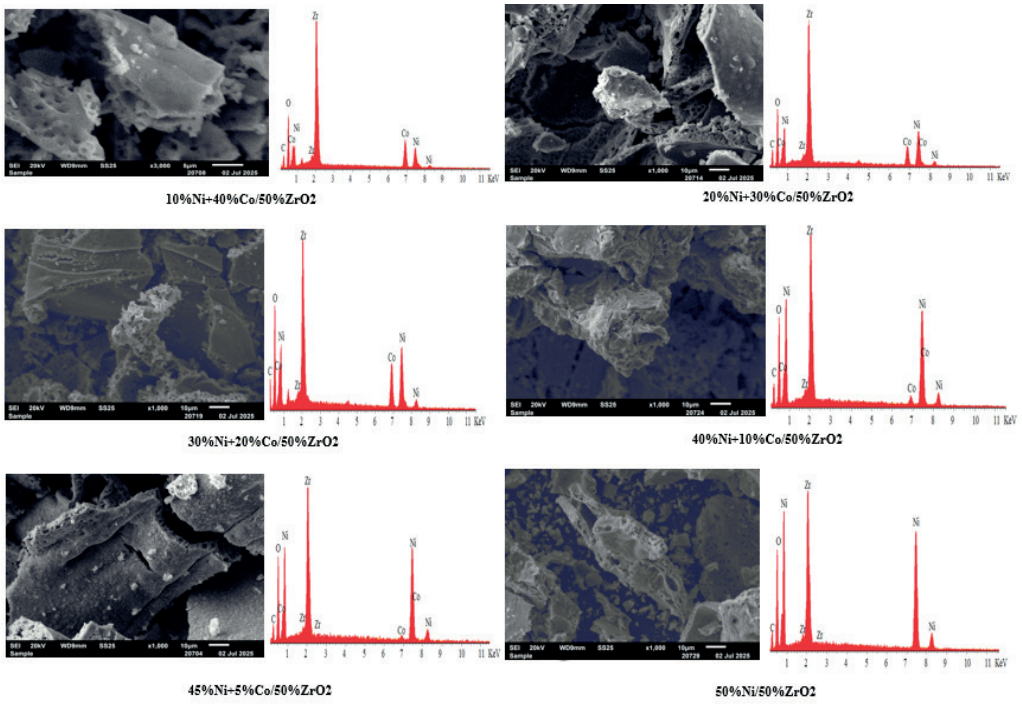

Figure 11 - Scanning electron micrographs of all fresh catalyst samples

Scanning Electron Microscopy analysis revealed significant morphological differences among the catalysts with varying Ni/Co ratios while maintaining a constant 50% Zr content. The catalyst with 45% Ni - 5% Co - 50% Zr exhibited a highly porous, sponge-like structure with uniform dispersion of metal particles, resulting from the efficient solution combustion synthesis process. In comparison, the 50% Ni - 50% Zr catalyst (without Co) showed larger aggregates and less uniform morphology, indicating that the addition of Co even in small proportions (5%) significantly improves structural homogeneity. The catalysts with intermediate compositions (10% Ni - 40% Co - 50% Zr, 20% Ni - 30% Co - 50% Zr, 30% Ni - 20% Co - 50% Zr, and 40% Ni - 10% Co - 50% Zr) demonstrated transitional morphological characteristics, where increasing Co content beyond 20% led to finer particle dispersion but reduced porosity.

Energy-Dispersive X-ray Spectroscopy mapping confirmed the homogeneous distribution of Ni, Co, and Zr in the 45% Ni - 5% Co - 50% Zr catalyst, with no evidence of metal segregation or large aggregates. This homogeneous distribution is critical for creating active sites accessible to reactants during catalytic processes. In contrast, the 50% Ni - 50% Zr catalyst showed zones of Ni-rich and Zr-rich phases, indicating incomplete mixing during synthesis. The catalysts with higher Co content (e.g., 10% Ni - 40% Co - 50% Zr) exhibited Co clustering, which may explain the reduced catalytic efficiency despite the finer particle size. The optimal dispersion in the 45% Ni - 5% Co - 50% Zr catalyst aligns with its superior performance in CO_2 hydrogenation, as reported in earlier studies.

Conclusion. In summary, the catalyst with the composition 45% Ni + 5% Co + 50% Zr, synthesized by the solution combustion method using urea, demonstrated high activity for the CO_2 hydrogenation reaction to methane. The optimal catalytic performance was achieved under the following conditions: a feed gas composition of 76% H_2, 19% CO_2, and 5% Ar; a space velocity of 24,000 h^{-1}; and a temperature of 300°C. Under these conditions, the process yielded a remarkable methane concentration of 98,8 - 99,2% with a selectivity of up to 97%. The high activity is attributed to the formation of simple oxides, as well as to metal particles within the catalyst structure, which actively promote the reaction. Furthermore, the solution combustion synthesis method offers significant practical advantages, including shorter preparation time, reduced economic cost, and enhanced environmental friendliness due to lower emissions during production. These benefits make SCS a highly promising technique for future catalytic applications.

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