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DRY REFORMING AND OXIDATIVE CONVERSION OF METHANE TO SYNTHESIS GAS IN THE PRESENCE OF Ni-Co-Mg-Ce CATALYSTS

Abstract. Catalytic carbon dioxide conversion of methane is the preferred method for producing synthesis gas in connection with the implementation of the process under milder conditions and solving environmental problems associated with a decrease in the rate of global warming. The activity of catalysts based on Ni-Co-Mg-Ce, obtained during combustion in solution, was studied in the processes of carbon dioxide conversion and oxidative conversion of methane. Comparative results indicate that the preparation of the catalyst by the impregnation method is inferior to the SHS method, in which higher values of the feed conversion and H₂ yield are obtained.

Key words: dry reforming, self-propagating high-temperature synthesis, methane, synthesis gas.

Introduction

Over the past two decades, the catalytic carbon dioxide conversion of methane to synthesis gas has attracted increasing attention as an alternative way for obtaining of synthesis gas (CO + H₂) by reaction $\text{CH}_4 + \text{CO}_2 \rightarrow 2\text{CO} + 2\text{H}_2$ [1-9]. This process is an attractive industrial process because of the low ratio of H₂/CO products and is therefore preferable for the Fischer-Tropsch process. According to the analysis of the energy prospects of the International Energy Agency (IEA) in 2013, "natural gas will certainly play a central role in meeting global energy needs, at least in the next two and a half decades." This means that technologies based on methane will have priority in the future.

Since 1950, methane production has doubled and could double by 2050. Annually 350-500 million tons of methane is added to the air (livestock, coal mining, oil and natural gas production, rice cultivation, garbage in landfills). Global annual emissions of anthropogenic greenhouse gases (by CO₂) are estimated at around 75%, and methane - around 14%. Reducing emissions of CH₄ and CO₂ can reduce global warming, reducing the risk of climate change. One way of doing this is catalytic reforming of methane with carbon dioxide to produce synthesis gas.

Many researchers have found that catalysts based on Fe, Co and Ni have a high activity, but one of the main problems arising during the application of this process is deactivation of catalyst mainly due to the deposition of carbon. Over the past decades, nickel catalysts have been extensively studied [10-12], a few studies have been done on cobalt catalysts, Ni-Co catalysts [13,14], and Ni-Ce-based catalysts [15-20]. It is known that the catalysts detected during these works show high activity and excellent anti-soot-forming properties.

At present, new composite catalysts are prepared not only by traditional methods of impregnation, but also by modern methods of self-propagating high-temperature synthesis (SHS) and combustion in solution [21-25]. Such conditions for the preparation of samples often lead to the synthesis of very active and selective catalysts. In this paper, a comparative study of the catalysts prepared by the above indicated methods in the processes of carbon dioxide conversion and oxidative conversion of methane is presented.

Experimental

Catalyst preparation

The SHS catalysts on the base of Ni-Co-Mg-Ce were prepared from powder mixtures. The specimens were preheated in an electric furnace at 700 – 900°C for several minutes. The second series of catalysts was prepared by the traditional method: samples were prepared by the incipient wetness impregnation of dispersed Al₂O₃ (granule size 100 – 200 μm, S – 57.7 m²/g) by water solutions of metal nitrates with subsequent heating on air at 250°C within 5 h and 600°C within 2 h.

Characterization techniques

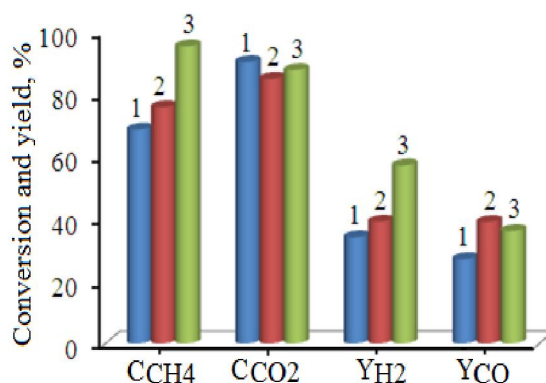
The analysis of the initial mixture and reaction products was carried out using a chromatograph "Chromos GC-1000" with the "Chromos" software and on a chromatograph "Agilent Technologies 6890N" (USA) with computer software. Chromatograph "Chromos GC-1000" is equipped with packed and capillary columns. The packed column is used for the analysis of H₂, O₂, N₂, CH₄, C₂H₆, C₂H₄, C₃-C₄ hydrocarbons, CO and CO₂. A capillary column is used to analyze of liquid organic substances, such as alcohols, acids, aldehydes, ketones and aromatic hydrocarbons. The temperature of the detector by thermal conductivity – 200°C, evaporator temperature – 280°C, column temperature – 40°C. Carrier gas velocity Ar = 10 ml/min. The chromatographic peaks were calculated from the calibration curves plotted for the respective products using the "Chromos" software for pure substances. Based on the measured areas of the peaks corresponding to the amount of the introduced substance, a calibration curve $V = f(S)$ was constructed, where V - amount of substance in ml, S - peak area in cm². Concentrations of the obtained products were determined on the basis of the obtained calibration curves. The balance of regulatory substances and products was ± 3.0%.

Catalytic reaction

Experiments on the oxidative conversion were carried out on flow type installation at atmospheric pressure in a tubular quartz reactor with a fixed catalyst bed. The catalyst was placed in the central part of reactor and quartz wool placed above and below the catalyst bed. The free space of the reactor below the catalyst bed was filled with quartz wool to reduce the free volume and gas phase zone of catalyst bed as well as to minimize the further reaction of the desired product in post-catalytic volume. Gas mixture containing methane and carbon dioxide (or oxygen) diluted with an inert gas (Ar) in the presence or absence of water vapor was used for oxidation.

Results and discussion

Dry conversion of methane was studied on the following catalyst 23% Ni + 13% Co + 9% Mg + 5% Ce/36.15% Al + 1.85% Mg, obtained by the SHS method at GHSV = 860 h⁻¹ and a ratio components of the reaction mixture CH₄ : CO₂ : Ar = 33.3 : 33.3 : 33.3 as well as at GHSV = 2500 h⁻¹ at a mixing ratio of CH₄ : CO₂ : Ar = 46.7 : 23.3 : 30.0. The studies were carried out both in the absence and in the presence of water vapor in the reaction mixture. The results are shown in Figure 1.



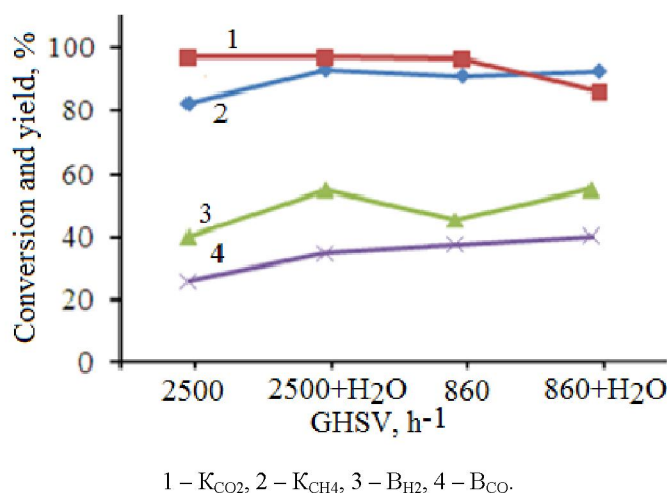
1 – 2500 h⁻¹ in the absence of water vapor, 2 – 860 h⁻¹ in the presence of water vapor, 3 – 860 h⁻¹ in the presence of water vapor.

Figure 1 - Influence of space velocity and the addition of water vapor on feed conversion and yield of reaction products

It can be seen from the figure the space velocity decreases from 2500 h^{-1} to 860 h^{-1} , the conversion of CH_4 , yields of H_2 and CO increase, and the addition of water vapor further increases the conversion of raw materials and the yield of H_2 .

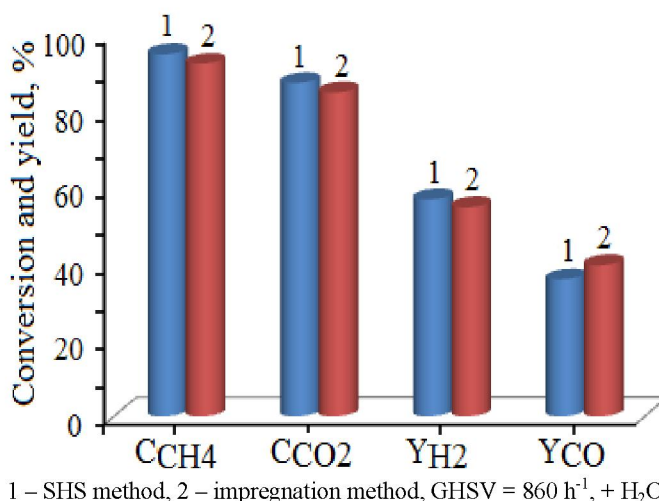
Similar experiments were carried out on the same catalyst composition, however, prepared by impregnation at space velocities of 860 and 2500 h^{-1} at 850 and 900°C in the presence and absence of water vapor. It can be seen from Figure 2 that, in the same way as in the case of SHS catalysts, the addition of water vapor favored the selective yield of H_2 and CO , with higher yields obtained at a space velocity of 860 h^{-1} .

The comparative results shown in Figure 3 indicate that the preparation of the catalyst by the impregnation method is inferior to the SHS method, in which higher values of the feed conversion and H_2 yield are obtained.



1 - K_{CO_2} , 2 - K_{CH_4} , 3 - B_{H_2} , 4 - B_{CO} .

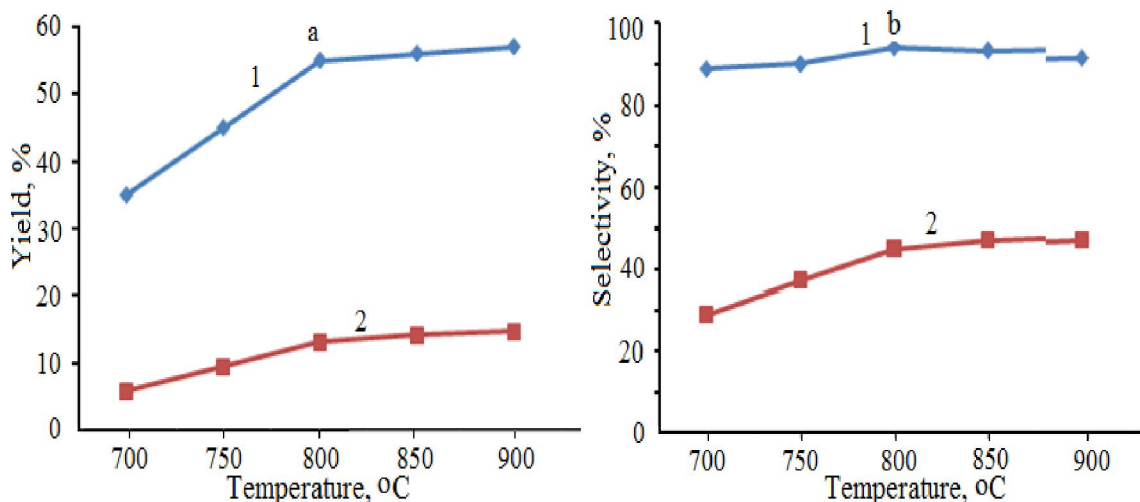
Figure 2 - Dependence of the conversion of raw materials and the yield of reaction products on the conditions of carrying out the process of carbon dioxide conversion on a catalyst prepared by impregnation



1 - SHS method, 2 - impregnation method, GHSV = 860 h^{-1} , + H_2O .

Figure 3 - Effect of the method of preparation of the catalyst on the parameters of the process of carbon dioxide conversion

Investigation of the activity of catalysts based on the initial mixture of $43\% \text{ Ni}(\text{NO}_3)_2 + 4\% \text{ Co}(\text{NO}_3)_2 + 2\% \text{ Mg}(\text{NO}_3)_2 + 1\% \text{ Ce}(\text{NO}_3)_2 + 50\% \text{ glycine} + 36.15\% \text{ Al}(\text{NO}_3)_3 + 11.85\% \text{ Mg}(\text{NO}_3)_2$ obtained in solution combustion synthesis (SCS) was conducted in the oxidative conversion of methane. A mixture of $34\% \text{ CH}_4$, $17\% \text{ O}_2$ and $50\% (\text{Ar} \pm \text{H}_2\text{O})$, $\text{CH}_4: \text{O}_2 = 2 : 1$ was used to study the oxidation of methane to synthesis gas in the temperature range $700 - 900^\circ\text{C}$. The results on the yield of hydrogen and CO , as well as the selectivity for the target products for these catalysts as a function of temperatures ($700 \pm 900^\circ\text{C}$) are shown in Figures 4a and 4b.



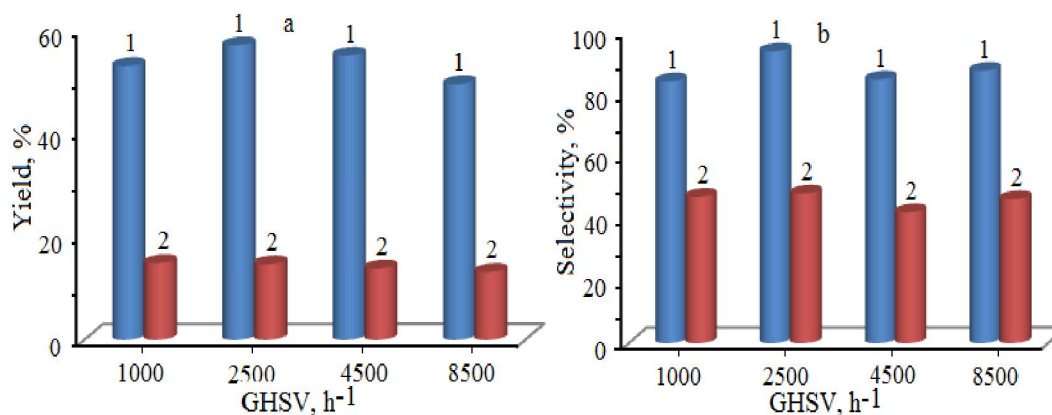
a – yield: 1 - H₂, 2 - CO; b – selectivity: 1 - H₂, 2 - CO; GHSC = 2500 h⁻¹.

Figure 4 - Effect of varying the process temperature on the yield and selectivity for H₂ and CO on the catalyst 43% Ni(NO₃)₂ + 4% Co(NO₃)₂ + 2% Mg(NO₃)₂ + 50% glycine + 36.15% Al(NO₃)₃ + 13.85% Mg(NO₃)₂

As a result of the temperature variation, it was found that carrying out the process at 900°C makes it possible to achieve the highest values of both yield and selectivity for H₂ and CO, but the ratio H₂/CO decreases from 6.1 (T = 700°C) to 3.9 (T = 900°C) at GHSV = 2500 h⁻¹.

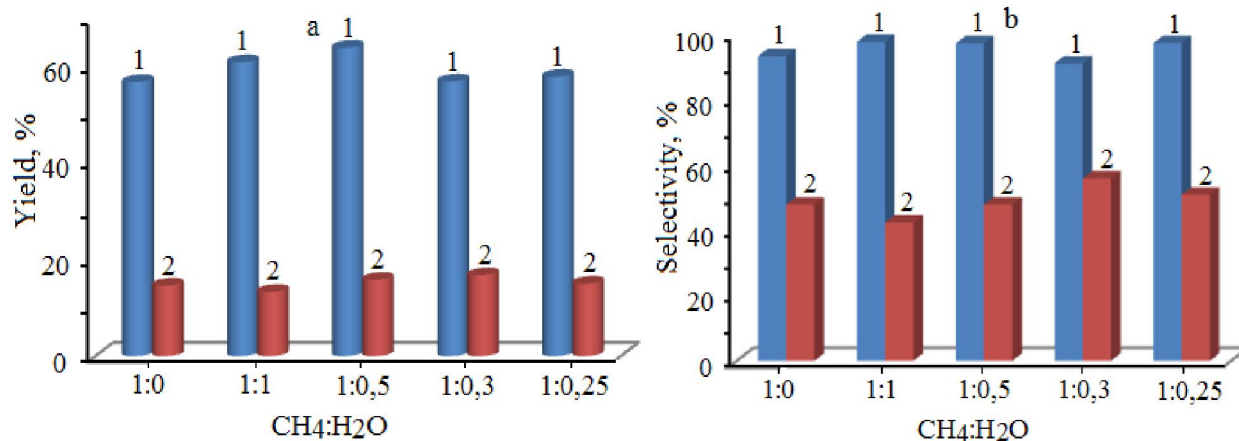
On this catalyst, the yields of hydrogen and CO, their selectivity as well as the H₂/CO ratio, which plays an important role in further syntheses of alcohols and hydrocarbons, were determined when the temperature and space velocities varied from 1000 to 8500 h⁻¹. It was shown it is possible to obtain the highest yield and selectivity parameters for the target products at space velocities from 1000 to 2500 h⁻¹. For example, up to 55.8 - 56.9% H₂, 14.6% CO with a selectivity up to 93.6% by hydrogen and 48% by CO, a ratio of H₂/CO = 3.8 - 3.9 were obtained at GHSV = 2500 h⁻¹. Similar results were obtained at a space velocity of 1000 h⁻¹. A further increase in the space velocity leads to a decrease in the process indices, Figures 5a and 5b.

On these catalyst compositions, the addition of water vapor to the initial reaction mixture and variation of CH₄ : H₂O ratio (1 : 0, 1 : 1, 1 : 0,5, 1 : 0,3, 1 : 0,25) were investigated. It has been established that the addition of water vapor to the CH₄ : H₂O = 1 : 0.5 ratio slightly increases the yield of synthesis gas, while increasing the yield of H₂ from 55.8 - 56.9 to 63.0 - 64.0%, and CO - from 14.0 to 15.6 - 15.8%, Figure 6. Further increase in the vapor content to the ratio CH₄ : H₂O = 1 : 1 leads to a decrease in yield and selectivity. The ratio of H₂/CO remains practically unchanged in the range 3.9 - 4.0.



a – yield: 1 - H₂, 2 - CO; b – selectivity: 1 - H₂, 2 - CO; GHSV = 2500 h⁻¹.

Figure 5 - Influence of the variation of the process space velocity on the yield and selectivity for H₂ and CO on the catalyst 43% Ni(NO₃)₂ + 4% Co(NO₃)₂ + 2% Mg(NO₃)₂ + 50% glycine + 36.15% Al(NO₃)₃ + 13.85% Mg(NO₃)₂



a – yield: 1 - H₂, 2 – CO; b – selectivity: 1 - H₂, 2 - CO; GHSV = 2500 h⁻¹.

Figure 6 - Effect of water vapor content in the reaction mixture on the yield and selectivity of the process for H₂ and CO

Reducing the dilution of the reaction mixture (as a result of decreasing the Ar content) increases the yields for H₂ (from 55.8 to 69.7%) and CO (from 14.0% to 21.0%), but decreases the selectivity for products (H₂ - up to 53.7%, CO - 32.5%) while maintaining the ratio H₂ : CO = 3.3.

Conclusion

23% Ni + 13% Co + 9% Mg + 5% Ce/36.15% Al + 1.85% Mg catalysts prepared by self-propagating high-temperature synthesis and impregnation method were investigated in dry conversion and oxidation of methane at different space velocities, temperatures and compositions of reaction mixture. It was found that the preparation of the catalyst by impregnation method is inferior to the SHS method, in which higher values of the feed conversion and H₂ yield are obtained. As a result of the temperature variation, it was found that carrying out the process at 900°C makes it possible to achieve the highest values of both yield and selectivity for H₂ and CO, but H₂/CO ratio decreases from 6.1 (T = 700°C) to 3.9 (T = 900°C) at GHSV = 2500 h⁻¹. It has been established that the addition of water vapor to the CH₄ : H₂O = 1 : 0.5 ratio slightly increases the yield of synthesis gas, while increasing the yield of H₂ from 55.8 - 56.9 to 63.0 - 64.0%, and CO - from 14.0 to 15.6 - 15.8%.

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МЕТАНДЫ СИНТЕЗ-ГАЗҒА Ni-Co-Mg-Ce КАТАЛИЗАТОРЛАРЫ ҚАТЫСЫНДА ҚҰРҒАҚ РЕФОРМИНГЛЕУ МЕН ТОТЫҚТЫРУ КОНВЕРСИЯСЫ

Аннотация. Жаһандық жылынуды төмендету жағдайларына байланысты экологиялық мәселелерді шешуде метаннан синтез-газ алу әдісінің оңтайлы үрдісін қолайлы жағдайда жүзеге асыру үшін каталитикалық көмірқышқылды конверсиясы тиімді болып табылады. Ертінде жану үрдісі арқылы алынған

Ni-Co-Mg-Ce негізіндегі катализаторлардың белсенділігі метанды тотықтыра айналдыру және көмірқышқылды конверсиясы үрдісінде зерттелді. Салыстырмалы зерттеу нәтижелерінің қорытындысы сіңіру әдісіне қарағанда өздігінен жану әдісімен дайындалған катализаторларда сутектің шығымы және шикізат конверсиясының мәні жоғары екенін көрсетті.

Түйін сөздер: құрғақ реформинг, жоғарытемпературада өздігінен жану синтезі, метан, синтез-газ.

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СУХОЙ РЕФОРМИНГ И ОКИСЛИТЕЛЬНАЯ КОНВЕРСИЯ МЕТАНА В СИНТЕЗ-ГАЗ В ПРИСУТСТВИИ Ni-Co-Mg-Ce КАТАЛИЗАТОРОВ

Аннотация. Каталитическая углекислотная конверсия метана является предпочтительным способом получения синтез-газа в связи с осуществлением процесса в более мягких условиях и решением экологических проблем, связанных со снижением темпов глобального потепления. Активность катализаторов на основе Ni-Co-Mg-Ce, полученных в процессе горения в растворе, была исследована в процессах углекислотной конверсии и окислительного превращения метана. Сравнительные результаты указывают на то, что приготовление катализатора методом пропитки уступает методу СВС, при котором получены более высокие значения конверсии сырья и выхода H₂.

Ключевые слова: сухой реформинг, самораспространяющийся высокотемпературный синтез, метан, синтез-газ.

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