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«Жанармай, катализ және электрохимия институты» АҚ

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НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Үлттық гылым академиясы «ҚР ¥ҒА Хабарлары. Химия және технология сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бүл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын үсынады. ҚР ¥ҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қогамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Socia1 Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество в глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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OXIDATIVE DIMERIZATION OF METHANE TO C_2 HYDROCARBONS

Abstract. The world's oil reserves are decreasing every day due to the continuous production and processing of the most modern technologies. Scientists all over the world are looking for various raw materials and methods to use the vast resources of natural gas as a substitute for petrochemicals. In this regard, great attention is drawn to natural gas as an alternative source of raw materials for petrochemical industries.

The purpose of this work is to study the reaction of methane dehydrogenation on new 20%La-10%Ce-20%Mg-50% glycine catalysts prepared by the SHS method to identify the optimal conditions for their preparation, concentration and ratio of metals, the influence of contact time and process temperature on the direction and mechanism of the reaction.

The results of the study of 20% La-10% Ce-20% Mg-50% glycine catalyst prepared by the SHS method in the process of oxidative dehydrogenation of methane into C_2 hydrocarbons are presented. On the basis of experimental studies, it was found that the composition of the catalyst exhibits high activity in the above reaction under the found optimal conditions.

Thus, the influence of reaction temperature on the developed composition of catalysts for oxidative conversion of methane has been determined that the optimum temperature for the selective formation of ethane and ethylene is T=700°C. It was found that for selective oxidation of a mixture of CH_4: O_2 : Ar in C_2 hydrocarbons the optimal conditions are: T=700°C, CH_4:O_2=2,5:1, 5000 h^{-1}.

Key words: ODM reaction, C_2 hydrocarbons, SHS method, temperature of reaction.

Introduction. The world's oil reserves are decreasing every day due to the continuous production and processing of the most modern technologies. Scientists all over the world are looking for various raw materials and methods to use the vast resources of natural gas as a substitute for petrochemicals. In this regard, great attention is drawn to natural gas as an alternative source of raw materials for petrochemical industries.

According to the annual Statistical Review of World Energy – 2018, published by British Petroleum (BP) at the end of 2017 proven natural gas reserves in the world are estimated at 193.5 trillion cubic meters.

It is known that natural gas is 90% methane. Of interest is the oxidative dimerization of methane into ethylene, which makes it possible to obtain a number of petrochemical products, such as polyethylene, polystyrene and many other chemical products. This is primarily due to the low cost of methane compared to other hydrocarbons [1].

Materials and methods. In the work of Karakaya [2] the $Mn/Na_2WO_4/SiO_2$ catalyst was studied for the oxidative dimerization of methane. The catalyst was prepared by impregnation methods. The influence of temperature, volume velocity and the ratio of reaction gases is investigated. It is determined that at a volume rate of 390 h-1 methane conversion is 38%. The maximum ethylene yield was 16% at 750°C and a gas ratio of $CH_4/O_2=2$.

In [3] the $Mn\text{-}Ce\text{-}Na_2WO_4/SiO_2$ catalysts prepared by the impregnation method were investigated for oxidative dimerization of methane. In particular, due to the double advantages of the tubular membrane reactor, a high methane conversion of 60.7% with selectivity of C_2+ 41.6%, ethylene/ethane ratio of 5.8% and ethylene yield of 19.4% at a space velocity of GHSV= 6050 $ml\cdot g^{-1}\cdot h^{-1}$ was achieved.

It is known that combustion in the solution is a new, quick and easy process for the synthesis of composite materials used in various processes. The main advantage of this method is the saving of energy and time, as well as the presence of self-propagating exothermic redox reactions between organic salts and fuel by mixing in an aqueous solution leading to the formation of nanocrystalline and high-purity solid nanomaterials [4-6].

In [7] Sr-Al, La-Sr-Al and Na_2WO_4–Mn/SiO_2 series of catalysts are prepared by solution combustion synthesis (SCS). The activity of catalysts was investigated at 450-850°C, the linear velocity of gases was 50 ml/min, the ratio of gases $CH_4:O_2:N_2$ = 32:8:10. During the experiments it was determined that in the temperature range 450-600°C catalysts are not active. The Sr-Al series of catalysts, Sr/Al=1.25 were active. On the catalyst Sr / Al=1,25 it was found that the maximum yield of C_2 hydrocarbons was 11.5% at~800°C ratio C_2H_4/C_2H_6 = 4,5. For each La-Sr-Al catalyst series, the C_2 yield and ethylene/ethane ratio are measured over a wide temperature range. Of the series of catalysts, the highest ethylene yield of 12.3~13.0% is achieved at 720°C, and the ratio of ethylene to ethane is 1.7 and 1.6, respectively. It was found that from the series of developed catalysts the highly active and selective catalyst is 10% Na_2WO4–5% Mn/SiO_2 providing the maximum yield of C_2 ~21% and the ethylene/ethane ratio~2, at 750°C.

Catalytic oxidative dimerization of methane on Ce-promoted catalysts Na_2WO_4/SiO_2 was carried out at atmospheric pressure in a micro quartz tubular reactor with a fixed bed. The highest methane conversion (50.1%) and C_2 yield (27.6%) were obtained on a catalyst with 3 wt.% Ce/5 wt.% Na_2WO_4/TiO_2 at 800°C. With a space velocity of 10.000 $ml\cdot g^{-1}/h^{-1}$, 0.2 g catalyst, P = 1 atm, the developed 3 wt.% Ce/5 wt.% Na_2WO_4/TiO_2 catalyst showed the best performance and stability for up to 50 hours at 800°C, high selectivity and yield of C_2, 56.4% and 27.6%, respectively, was also shown [8].

However, we have previously shown that possibility of obtaining a number of products, such as oxygenates, olefins, synthesis gas and CO_2+H_2O in the process of oxidative conversion of light alkanes on different types of catalysts [9-11].

Preparation of catalysts by solution combustion synthesis (SCS) is one of the new directions in catalysis. SCS is based on an exothermic redox reaction between soluble salts with organic fuel in an aqueous solution. The effectiveness of this method lies in the simplicity and ease of preparation of catalysts, short reaction time and relatively low cost, and therefore the efficiency of the process. The SCS method is used in various industries, especially for the preparation of ceramic materials, intermetallics, catalysts, pigments, alloys, etc. [12]. SCS is a new modern type of self-propagating high-temperature synthesis (SHS) [13]. The basic principles of SHS can be summarized as follows: rapid auto wave combustion-like selfsustaining reactions yielding resultant products of desired composition and structure, complete or partial elimination of external energy supply by the utilization of the internal heat released in the chemical reactions, control of the process rate, temperature, degree of conversion, composition and structure of products by variation in the rates of heat release and transfer [14.15]. Very high interest in the SHS catalyst can be explained by their high activity and advantages of methods for preparation of catalysts by the SHS methods in comparison with the traditional ones. The method exploits self-sustaining solid-flame combustion reactions for the internal development of very high temperatures over very short periods. It therefore offers many advantages over traditional methods such as much lower energy costs, ease of manufacture and capability for producing materials with unique properties and characteristics. It was found, that method offers a good possibility for the preparation of new, active ceramic catalysts and carriers with compositions, structure and properties, which satisfy the stringent requirements of many applications [16]. Attractive for industrial production: much lower energy consumption than traditional production methods, much lower energy costs, possibility for "just-in-time" manufacturing, high productivity, cheap catalysts, relatively simple process - easily adaptable to industrial scale, controlled physicochemical properties of products, large range of new materials which can be used in catalysis, it has wide diapason of structural forms of products - from granules of different size to blocks of honeycomb structure and different geometric forms

A disadvantage of the catalysts for ethylene, prepared by SHS [7.9] is not sufficiently high catalyst activity, but it is not complete conversion of methane to ethylene and high temperature process.

The purpose of this work is to study the reaction of methane dehydrogenation on new 20%La-10%Ce-20%Mg-50% glycine catalysts prepared by the SHS method to identify the optimal conditions for their preparation, concentration and ratio of metals, the influence of contact time and process temperature on the direction and mechanism of the reaction.

Catalyst preparation

For the preparation of the catalyst, certain amounts of nitrates of the corresponding salts are weighed. These salts are pre-ground in an agate mortar and then mixed in a porcelain dish. Then 10 ml of distilled water is gradually added to this mixture of salts, the mixture is stirred in air for several minutes until complete dissolution.

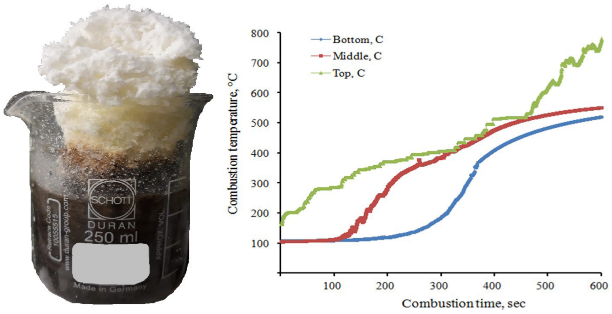

Figure 1 - 20% La-10% Ce-20% Mg-50% glycine catalyst prepared by SHS method and change of temperature-time profile during catalyst preparation

Pre-included muffle furnace to the required temperature (in our case, up to 500°C). The prepared mixture from a porcelain cup is transferred to a 200 ml heat-resistant glass beaker and placed in a heated muffle furnace. After 2-3 minutes with an incomplete opening of the door of the muffle furnace, it is visually possible to observe burning in the solution, at which this mixture rises along the walls of the glass during rapid boiling. Urea and glycine are added to the composition of SHS catalysts to improve the combustion process. The presence of glycine in the composition of the catalyst contributes to a change in the color of the solution in brown color during combustion. Then the glass is cooled in air and the finished catalyst is placed in glass cups.

Characterization techniques

Analysis of the initial mixture and the reaction products was performed using "Chromos GC-1000" (Russia) chromatograph, which was equipped with packed and capillary columns. The packed column is used for the analysis of H_2, O_2, N_2, CH_4, C_2H_6, C_2H_4, C_3-C_4 hydrocarbons, CO and CO_2. A capillary column is used to analyze of liquid organic substances, such as alcohols, acids, aldehydes, ketones and aromatic hydrocarbons. Temperature of the detector by thermal conductivity – 200°C, evaporator temperature – 280°C, column temperature – 40°C. The speed of the carrier gas Ar is 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 cm_2. 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%.

Physico-chemical research

The specific surface area and measurement of the pore distribution of the developed catalysts were studied by the BET method (Bronauer-Emmett-Teller) on a GAPP V-Sorb 2800 analyzer (China). Nitrogen with helium was used as carrier gas. BET method was carried out at Advanced Ceramics and Composites Laboratory, Institute of Nanoscience and Nanotechnology NCSR “Demokritos” (Athens, Greece). Nitrogen with helium was used as carrier gas.

Results and discussion

Table 1 shows data on the study of the specific surface of La-Ce-Mg-glycine catalysts prepared by the SHS method. From the data of the table it can be seen that a series of catalysts was prepared with a variation in the percentage of the active phase consisting of La, Ce and Mg deposited on the carrier 50% glycine. The surface area calculated by BET varied in the range from 9.42 to 24.96 m^2/g, and when calculated by Langmuir varied in the range from 12.45 to 35.25 m^2/g.

Table 1 - Change of specific surface of La-Ce-Mg-glycine catalysts prepared by SHS method

Catalysts	Multi-BET Area (m^2/g)	Langmuir Area (m^2/g)
30%$Ce(NO_3)_2$+ 20% $Mg(NO_3)_2$ +50% glycine	24.96	35.25
10%$La(NO_3)_3$ + 20%$Ce(NO_3)_2$+ 20% $Mg(NO_3)_2$ +50% glycine	22.87	32.19
15%$La(NO_3)_3$ + 15%$Ce(NO3)_2$+ 20% $Mg(NO_3)_2$ +50% glycine	18.39	25.48
20%$La(NO_3)_3$ + 10%$Ce(NO_3)_2$+ 20% $Mg(NO_3)_2$ +50% glycine	9.42	12.45
30%$La(NO_3)_3$ + 20% $Mg(NO_3)_2$ +50% glycine	21.91	30.56

It is known from the literature that SHS catalysts do not have a large surface, because in the case of using SHS catalysts in the reaction mixture in the gas phase, the entire volume of the catalyst works, not only its surface.

In the first series of experiments, the effect of reaction temperature on the yield of products was investigated on the developed 20%$La(NO_3)_3$+10 %$Ce(NO_3)_2$+20%$Mg(NO_3)_2$+50% glycine catalyst at oxidation of 43% CH_4+17% O_2+40% Ar at 5000 h^{-1}. Figure 2 shows that the formation of ethane and ethylene in the reaction mixture is observed in the temperature range from 600 to 900°C. 12.7% of ethylene is formed at a temperature of 600°C and increases to 13.5% at 700°C, but then there is a decrease to 0.5% with an increase in temperature to

900°C. Thus, the optimal temperature for producing 13.5% ethylene and 6.5% ethane is T=700°C.

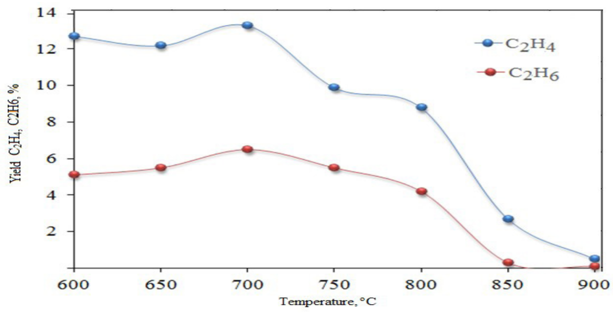

Figure 2 - Investigation of influence of reaction temperature on the yield of C_2H_4 and C_2H_6

It is known that the most thermally stable of alkanes is methane (destruction begins above 560°C), mainly due to the absence of C-C-bonds, less strong than C-H bonds. The process of ODM was carried out when the volume velocity varied from 2500 to 7500 h^{-1} in the temperature range from 600 to 900°C. As can be seen from the data of figure 3, when the volume velocity changes, the conversion of methane increases with an increase in temperature from 12 to 37.5%. It was found that the most optimal conversion rate of the initial methane is the volume velocity of 2500 h-1 compared to the data obtained at the volume velocity of 5000 and 7500 h^{-1}. It is determined that a sharp increase in conversion at temperatures 850 and 900°C is associated with the formation of hydrogen and CO in the reaction mixture.

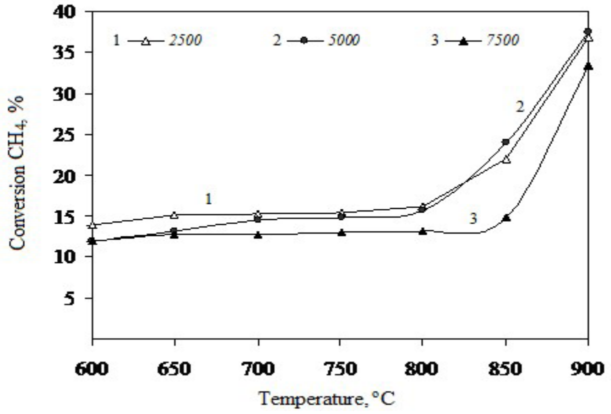

Figure 3 - Influence of volume velocity on the activity

of 20%La+10%Ce+20%Mg+50% glycine catalyst

Figure 4 presents data on the study of the effect of varying the volume velocity of the reaction on the yield of ethylene with a change in the reaction temperature from 600 to 900°C. As can be seen from the data of figure 4, the formation of 14.5-14.8% of ethylene passes through the maximum at T=700°C at volume velocity of 2500 and 5000 h^{-1}.

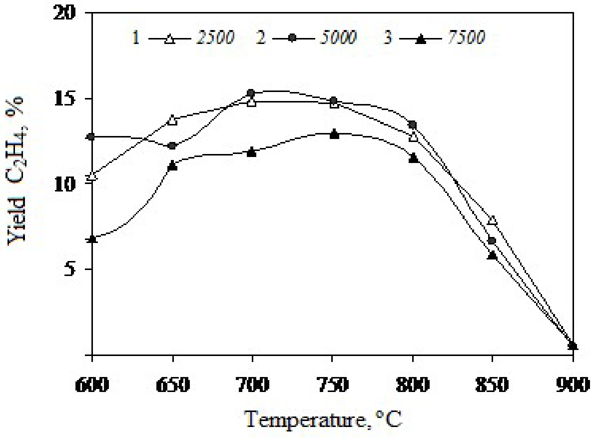

Figure 4 - Influence of volume velocity on the yield of ethylene

Thus, the study of the activity of the developed catalyst in the reaction of ODM showed that the optimal volume velocity of the process is 2500 and 5000 h^{-1}.

In the study of the activity of 20%La-10%Ce-20%Mg-50% glycine catalyst in the ODM into ethylene, the ratio of reaction gases CH_4: O_2 : Ar (%) was varied in the following ratios 2/1 (34:17:49), 2,5/1 (43:17:40), 3/1 (51:17: 32), 4/1 (68:17:15) and 5/1 (83: 17: 0). It was found that the optimal ratio of reaction gases CH4: O2 for the formation of ethylene is the ratio CH_4: O_2 = 2.5/1 at which starting from the temperature of 600°C the yield of ethylene gradually increases from 10.5 to 14.8% and passes through the maximum, then again decreases to 0.6% with increasing temperature (figure 5). Further on the activity indicator the ratio CH_4: O_2 = 2/1 is located, where it is found that the higher the ratio of reaction gases in the reaction mixture, the lower the yields of the target product.

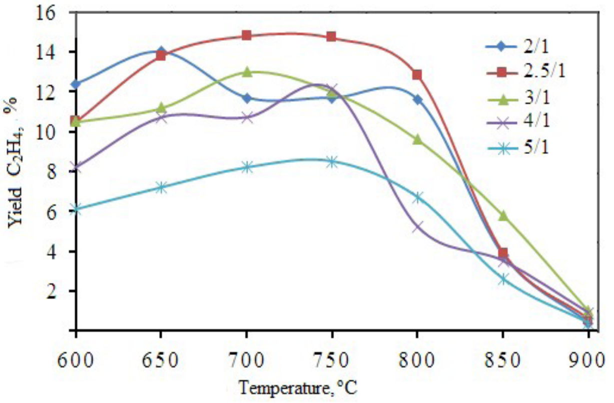

Figure 5 - Change of the yield of C_2H_4 at varying the ratio of reaction gases

This is explained by the fact that the methane molecule is thermodynamically stable and an increase in the reaction mixture of methane negatively affects the activity of the catalyst, and dilution of the reaction mixture with an inert gas argon positively affects the activity of the catalyst.

Figure 6 shows the data of changes in the specific surface area and measurement of the pore distribution of catalysts studied by the BET method.

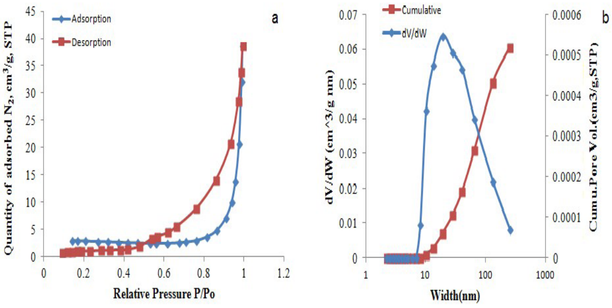

Figure 6 - Investigation of the isotherm (a) and pore distribution (b) on the surface of 20%La$(NO_3)_3$+10 %Ce$(NO_3)_2$+20%Mg$(NO_3)_2$+50% glycine catalyst

In [17] the authors give 5 types of possible hysteresis. For the developed 20%La$(NO_3)_3$+10% Ce$(NO_3)_2$+20%Mg$(NO_3)_2$+50% glycine catalyst corresponds to the hysteresis type A cylindrical shape.

As for the pore size, figure 6 shows that the size and uniform distribution of pores occurs mainly in the range from 3 to 10 nm, as well as from 10 to 100 nm, i.e. both small and large active particles are present in the catalyst.

Conciusion. Thus, the influence of reaction temperature on the developed composition of catalysts for oxidative conversion of methane has been determined that the optimum temperature for the selective formation of ethane and ethylene is T=700°C. It was found that for selective oxidation of a mixture of CH_4: O_2 : Ar in C_2 hydrocarbons the optimal conditions are: T=700°C, CH_4: O_2 = 2,5/1, 5000 h^{-1}.

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МЕТАННЫҢ C_2 КӨМІРСУТЕКТЕРІНЕ ДЕЙІН ТОТЫҒУ ДИМЕРИЗАЦИЯСЫ

Аннотация. Мұнайдың әлемдік қорлары ең заманауи технологиялар бойынша үздіксіз өндіру мен өңдеудің арқасында күн сайын қысқаруда. Дүние жүзіндегі ғалымдар мұнай химиясын алмастырғыш ретінде үлкен табиғи газ ресурстарын пайдаланудың әртүрлі шикізаттары мен әдістерін іздейді. Осыған байланысты мұнай-химия өнеркәсібі үшін шикізаттың балама көзі ретінде табиғи газға көп көңіл бөлінеді.

Бұл жұмыстың мақсаты жаңа 20%La-10%Ce-20%Mg-50% глицин катализаторларында метанның дегидрлеу реакциясын зерттеу, оларды өндірудің оңтайлы жағдайларын, металдардың концентрациясы мен қатынасын, жанасу уақыты мен температураның реакция бағыты мен механизміне әсерін анықтау.

20%La-10%Ce-20%Mg-50% C_2 көмірсутектеріне метанның тотығу дегидрлеу процесінде СВС әдісімен алынған глицин катализаторын зерттеу нәтижелері ұсынылған. Эксперименттік зерттеулер негізінде катализатордың құрамы табылған оңтайлы жағдайларда жоғарыда көрсетілген реакцияда жоғары белсенділік танытатыны анықталды.

Осылайша, реакция температурасының метанның тотығу конверсиясы катализаторларының дамыған құрамына әсері этан мен этиленнің селективті түзілуі үшін оңтайлы температура Т=700°С екендігі анықталды. C_2 көмірсутектеріндегі CH_4: O_2: Ar қоспасының селективті тотығуы үшін оңтайлы жағдайлар мыналар болып табылады: T=700°C, CH_4: O_2 = 2,5:1, 5000 $сағ^{-1}$.

Түйін сөздер: МТД реакциясы, C_2 көмірсутектері, ӨЖЖТС әдісі, реакция температурасы.

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ОКИСЛИТЕЛЬНАЯ ДИМЕРИЗАЦИЯ МЕТАНА ДО C_2 УГЛЕВОДОРОДОВ

Аннотация. Мировые запасы нефти сокращаются с каждым днем из-за непрерывной добычи и переработки по самым современным технологиям. Ученые всего мира ищут различные виды сырья и методы использования огромных ресурсов природного газа в качестве заменителя нефтехимии. В связи с этим большое внимание уделяется природному газу как альтернативному источнику сырья для нефтехимической промышленности.

Целью данной работы является изучение реакции дегидрирования метана на новых 20% La-10% Ce-20% Mg-50% глицин катализаторах, полученных методом СВС, с целью выявления оптимальных условий их получения, концентрации и соотношения металлов, влияния времени контакта и температуры процесса на направление и механизм протекания реакции.

Представлены результаты исследования 20% La-10% Ce-20% Mg-50% глицинового катализатора, полученного методом СВС в процессе окислительного дегидрирования метана в C_2 углеводороды. На основании экспериментальных исследований установлено, что состав катализатора проявляет высокую активность в вышеуказанной реакции при найденных оптимальных условиях.

Таким образом, по влиянию температуры реакции на разработанный состав катализаторов окислительной конверсии метана установлено, что оптимальной температурой для селективного образования этана и этилена является $T=700^oC$. Установлено, что для селективного окисления смеси CH_4: O_2: Ar в C_2 углеводороды оптимальными условиями являются: $T=700^oC$, $CH_4:O_2=2,5:1$, объемная скорость 5000 $ч^{-1}$.

Ключевые слова: реакция ОДМ, C_2 углеводороды, СВС-метод, температура реакции.

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ҰНДЫ КОНДИТЕР ӨНДІРІСІНДЕ ДӘСТҮРЛІ ЕМЕС ШИКІЗАТТАРДЫ ҚОЛДАНУДЫҢ ТИІМДІЛІГІ

Аннотация. Қазіргі таңда тағам өндірісі, соның ішінде кондитерлік өнімдер, инновациялық технологиялардың ең көп таралған объектілері болып табылады. Кондитер өндірісі-тамақ өнеркәсібінің ең тұрақты салаларының бірі. Оларды тағамдық құндылығы жоғары өнімдермен байыту үшін, дәстүрлі емес шикізаттарды пайдаланады.

Тамақтану элементтерінің ішінде адам ағзасының өз ақуыздарын синтездеуге қажетті аминқышқылдарының көзі болып табылатын белоктар маңызды рөл атқарады. Ақуыздар-бұл барлық мүшелерді, гормондарды, ас қорыту шырындарын, ферменттерді және т.б. құрайтын пластикалық материал.

Диеталық тамақтануда талшықтар да маңызды. Олардың тамақтанудағы рөлі әртүрлі: олар жалған қанықтыру әсерін жасайды, асқазан қабырғасына әсер етеді. Ішектен өткен кезде диеталық талшықтар адсорбциялық қасиеттерді көрсетіп, суды сақтай алатын кесек түзеді, нәтижесінде токсиндердің, ауыр металдардың тұздарының, бактериялардың, вирустардың концентрациясы төмендейді, ішек қабырғасының рецепторлары тітіркенеді, ішек транзиті жеделдейді [1-3].

Мақалада дәстүрлі емес шикізат ретінде: қант қызылша ұнтағы, жасымық ұны алынды. Созылмалы печеньенің жаңа түрі мен жасалу технологиясы құрастырылды.

МАЗМҰНЫ – СОДЕРЖАНИЕ – CONTENTS

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