UDC 544.478-03

HYDROGEN-RICH GAS PRODUCTION BY CATALYTIC DECOMPOSITION OF OXYGENATED COMPOUNDS OF C_1 CHEMISTRY

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Received 02.10.2020
Accepted 13.12.2020

Abstract: *Catalytic decomposition of oxygenated compounds of C_1 chemistry into hydrogen rich gas was studied over Pt/CeO_2-ZrO_2 catalyst. In particular, formic acid, methanol, dimethyl ether and dimethoxymethane were decomposed under atmospheric pressure into hydrogen-rich gas at temperatures below 450 °C. Challenges and benefits of each reaction in producing hydrogen-rich gas for fuel cell feeding are discussed.*

Key words: *Catalytic decomposition, oxygenates, C_1 chemistry, formic acid, methanol, dimethyl ether, dimethoxymethane, platinum, hydrogen, synthesis gas*

1. Introduction

Ever-increasing role of modern electronic devices in the human life stimulates active research and development in low-power fuel cell based portable and autonomous power units [1, 2]. The fuel for such power units is hydrogen or hydrogen-rich gas, which can be produced by reforming hydrocarbons or oxygenates [3-6]. A literature survey of current studies shows that oxygenated compounds of C_1 chemistry, such as formic acid (FA) [7-9], methanol [10, 11], dimethyl ether (DME) [12,13] and dimethoxymethane (DMM) [14-17], can be easily converted into hydrogen-rich gas at relatively low temperatures as compared to conventional hydrocarbon fuels. In addition, these oxygenates, unlike hydrocarbon fuels are free of impurities such as sulfur compounds that is a poison for most metal catalysts. The last evidence means that no sulfur scrubbing reactor is required. Among the known processes for hydrocarbon catalytic conversion (steam reforming, partial oxidation and decomposition), decomposition is thought to be promising for creating a compact "fuel processor:" this process requires no water or air tanks and respective supply and flow control systems.

Based on the above facts and taking into account a good performance of Pt-containing catalyst in the decomposition of FA (1) [7], methanol (2) [10], DME (3) [12] and DMM (4) [15], as well as a lower cost of Pt as compared to Pd and Rh, we decided to perform comparative studies of catalytic decomposition of the mentioned oxygenated compounds into hydrogen rich gas over the Pt/CeO_2-ZrO_2 catalyst.

Overall FA and methanol decomposition reactions are expressed by equations as follows:

$$HCOOH = H_2 + CO_2 \qquad (1)$$
$$CH_3OH = 2H_2 + CO \qquad (2)$$

Overall DME and DMM decomposition reactions are most likely described by equations:

$$CH_3OCH_3 = H_2 + CO + CH_4 \qquad (3)$$
$$CH_3OCH_2OCH_3 = 2H_2 + 2CO + CH_4 \qquad (4)$$

Purpose of the manuscript

The present work reports on the performance of the Pt/CeO_2-ZrO_2 catalyst in FA, methanol, DME and DMM decomposition reactions into hydrogen-rich gas and provides a comparative analysis to elucidate the challenges and benefits of each reaction for producing hydrogen-rich gas for fuel cell feeding.

2. Experimental procedure

Pt/CeO_2-ZrO_2 catalyst (1.9 wt % Pt) was prepared by sorption-hydrolytic deposition as described in [17,18]. A solution of H_2PtCl_4 was mixed with Na_2CO_3 providing the molar ratio of Na/Cl=1. The obtained mixture was brought into contact with an aqueous suspension of the CeO_2-ZrO_2 powder (Ecoalliance Ltd. Russia). According to our previous work [18], CeO_2-ZrO_2 support had fluorite structure with crystallite particles of 10 nm in size; the size of Pt particles in the fresh catalyst was ~2 nm. The BET specific surface area of Pt/CeO_2-ZrO_2 was ~70 m/g.

Catalytic experiments on FA, methanol, DME and DMM decomposition were performed in a U-shaped quartz reactor (i.d. 6 mm) under atmospheric pressure. Prior to the catalyst testing, the Pt/CeO_2-ZrO_2 catalyst (0.25–0.5 mm) was reduced in situ at 400 °C for 1 h using 10 vol. % H_2/Ar with a total flow rate of 3000 mL/h. Then the catalyst was exposed to the feed composed of (vol. %): 10 HCOOH and 90 N_2 for FA decomposition; 10 CH_3OH and 90 N_2 for methanol decomposition; 10 $CH_3OCH_2OCH_3$ and 90 N_2 for DMM decomposition; 10 CH_3OCH_3 and 90 N_2 for DME decomposition. Total gas hourly space velocity (GHSV) was 10000 h^{-1}.

FA, methanol and DMM were introduced to the reactor by bubbling N_2 through a saturator filled with respective liquid compound. Bronkhorst mass flow controllers fed the DME, N_2 and H_2 (for catalyst reduction). The composition of the reagents and reaction products were evaluated by a gas chromatograph (GC Chromos-1000). The FA, methanol, DME and DMM conversions (X_i) and H_2 productivity ($W(H_2)$) were calculated using the following equations:

$$X_i(\%) = \frac{C_i^0 - C_i \times \dfrac{C_{N_2}^0}{C_{N_2}}}{C_i^0} \times 100 \qquad (5)$$

$$W_{H_2}\left(\frac{L}{g_{cat} \cdot h}\right) = \frac{F \cdot C_{H_2} \times \dfrac{C_{N_2}^0}{C_{N_2}}}{100 \cdot m_{cat}} \qquad (6)$$

where C_i^0 , $C_{N_2}^0$ are the inlet concentrations (vol.%) of oxygenate (FA, methanol, DME and DMM) and N_2;

C_i , C_{N_2}, C_{H_2} are the outlet concentrations (vol.%) of unconverted oxygenate, N_2, H_2.

F – total flow rate of the inlet reaction mixture (L/h); m_{cat} – catalyst weight (g).

The H_2 selectivity ($S(H_2)$) for decomposition reactions was calculated as total moles of H_2 actually produced through division by the moles of H_2 theoretically produced. The carbonaceous product selectivity (S) was defined as the amount of carbon in this product divided by total amount of carbon in converted compound of C1 chemistry.

To evaluate carbon formation in DMM and DME decomposition, the spent catalysts were studied by temperature-programmed oxidation (TPO) using a TG209F Libra Termo microbalance instrument (Netzch, Germany). The feed gas, 6 vol.% O_2/He, flowed at 4.2 L/h. The sample (~50 mg) was heated from 25 to 600 °C at 600 °C/h. The outlet CO_2 concentration was monitored on-line by a QMS-200 mass-spectrometer (Stanford Research Systems, USA). The amount of the coke deposited on the catalyst was determined from that of CO_2 released in the TPO runs.

3. Results and discussion

3.1. Catalytic decomposition of formic acid

Fig. 1 shows the effect of temperature on FA conversion and product selectivity (H_2, CO_2, CO and CH_4) in FA decomposition over Pt/CeO_2-ZrO_2 catalyst. As the reaction temperature increased from 150 to 400 °C, the FA conversion increased and reached ~100 % at ~200 °C. Hydrogen and carbon dioxide were the main reaction products (S > 80 %), suggesting that the FA decomposition predominantly proceeds according to equation (1). Note that the temperature dependencies of H_2 and CO_2 selectivities were similar: the maximum in both curves (~94 %) was observed at temperatures of 250-300 °C. Further decrease of the selectivities at temperatures above 300 °C is explained by intensification of side reactions (7) and (8) yielding CO and water:

$$HCOOH = CO + H_2O \quad (7)$$

$$CO_2 + H_2 = CO + H_2O \quad (8)$$

The observed temperature dependencies are in agreement with early published data on FA decomposition over Pd/ZnO catalyst [8]. At temperatures above 350 °C, negligible amount of methane was produced as a by-product by hydrogenation of carbon oxides. This observation is in agreement with the course of CO methanation reaction over Pt-containing catalyst [19,20]:

$$CO + 3H_2 = CH_4 + H_2O \quad (9)$$

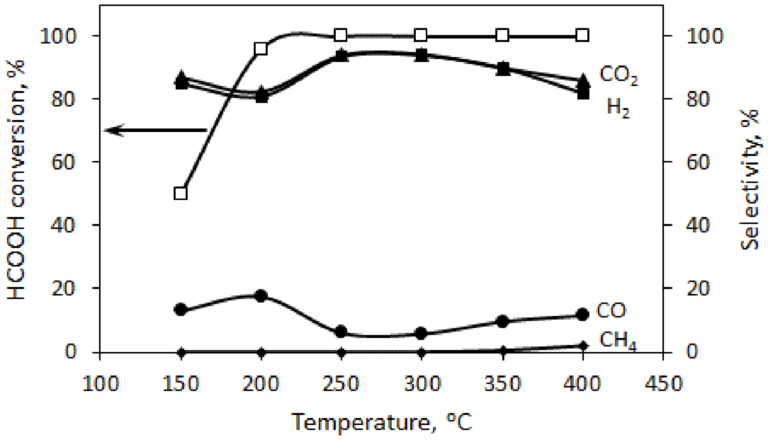

Fig.1. Effect of temperature on formic acid conversion and product selectivity (H_2, CO, CO_2, CH_4) in FA decomposition over Pt/CeO_2-ZrO_2 catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h^{-1}. Inlet composition: HCOOH:N_2 = 10:90 vol. %.

In general, the Pt/CeO_2-ZrO_2 catalyst demonstrates good performance for FA decomposition into hydrogen-rich gas. It provides 100% FA conversion with high (~ 94 %) H_2 and CO_2 selectivity at 250-300 °C. With consideration of the results obtained, we calculated the hydrogen-rich gas composition (excluding N_2) after the FA decomposition at 250-300 °C (vol. %): 47 H_2; 47 CO2; 3 H_2O and 3 CO. As stated in [21], a gas mixture of this composition can be used directly for feeding high-temperature polymer electrolyte membrane fuel cells (HT PEMFCs) as well as solid oxide fuel cells (SOFCs) without any cleaning processes.

3.2. Catalytic decomposition of methanol

Unlike formic acid, methanol decomposes over the Pt/CeO_2-ZrO_2 catalyst with the predominant formation of H_2 and CO. Fig. 2 shows temperature dependencies of methanol conversion and the selectivity of the main reaction products (H_2, CO, CO_2, CH_4) in methanol decomposition over Pt/CeO_2-ZrO_2 catalyst. At 200 °C, methanol conversion was ~20% which increased as temperature increased to reach ~100 % at 300 °C. H_2 and CO are main reaction products at 200–300 °C; their selectivity was ~100 % suggesting that the methanol decomposition over Pt/CeO_2-ZrO_2 proceeds according to reaction (10):

$$CH_3OH = 2H_2 + CO \quad (10)$$

The high reaction selectivity is consistent with the data reported in previously published works [10].

Then, as the temperature rose above 300 °C, the product distribution changed drastically; the H_2 and CO selectivity decreased, whereas the CH_4 and CO_2 selectivity increased up to 10 and 40 %, respectively. The formation of CH_4, CO_2 and H_2O (not shown in Fig. 2) is most likely related to consecutive proceeding of CO hydrogenation to methane and water (9) and Water Gas Shift (WGS) reaction (11):

$$CO + H_2O = H_2 + CO_2 \qquad (11)$$

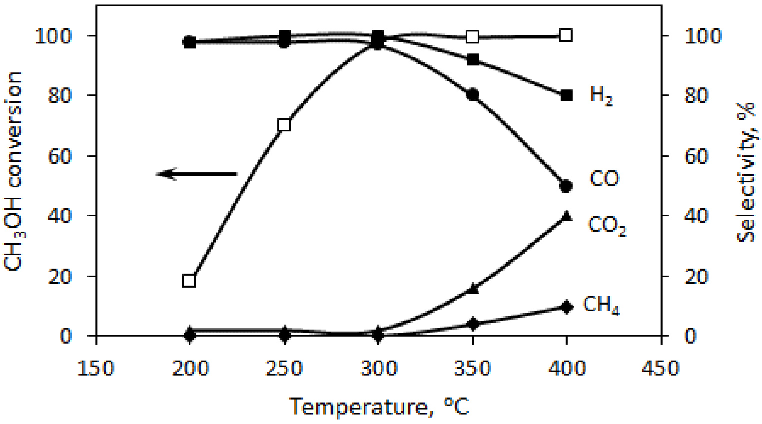

Fig. 2. Effect of temperature on methanol conversion and product selectivity (H_2, CO, CO_2, CH_4) in methanol decomposition over Pt/CeO_2-ZrO_2 catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h^{-1}. Inlet composition: CH_3OH:N_2 = 10:90 vol. %.

Thus, Fig. 2 data show that Pt/CeO_2-ZrO_2 is effective for methanol decomposition into hydrogen-rich gas. Moreover, the catalyst demonstrates good stability during the reaction. The results obtained prove that methanol decomposition over the Pt/CeO_2-ZrO_2 catalyst is quite promising for efficient production of synthesis gas for SOFC feeding applications.

3.3. Catalytic decomposition of DMM and DME

Fig. 3 illustrates the effect of temperature on the conversion and product selectivity in DMM and DME decomposition reactions over Pt/CeO_2-ZrO_2 catalyst. As is seen in Fig. 3, the DMM and DME conversions over Pt/CeO_2-ZrO_2 catalyst proceed in a higher temperature region as compared to FA and methanol decomposition. Moreover, the temperature dependencies of the conversion and product distribution for DMM decomposition were similar to those for DME decomposition. In particular, at 300 °C the DMM and DME conversions did not exceed 20%, increased with temperature, and reached ~100 % at temperatures above 400 °C yielding H_2, CO, CH_4 and CO_2 ~~were~~ as the main reaction products for both DMM and DME decomposition reactions. The selectivities of water, ethylene and methanol were negligibly low and are not shown in Fig. 3. At 300 °C, H_2 and CO were the main products ($S > 60$ %) of DMM and DME decomposition reactions. Then, as the reaction temperature increased, the product distribution changed strongly: H_2 and CO selectivity decreased from 60-70% to 20-40% with simultaneous increase in CH_4 formation up to 50-70 %. Note that the CO_2 selectivity increased as temperature rose, but didn’t exceed 15% even at 400-450 °C. Similarly to methanol decomposition (Fig. 2), the regularities of product distribution presented in Fig. 3 can be associated with the formation of CH_4 and CO_2 by, for example, side-reactions (9) and (11).

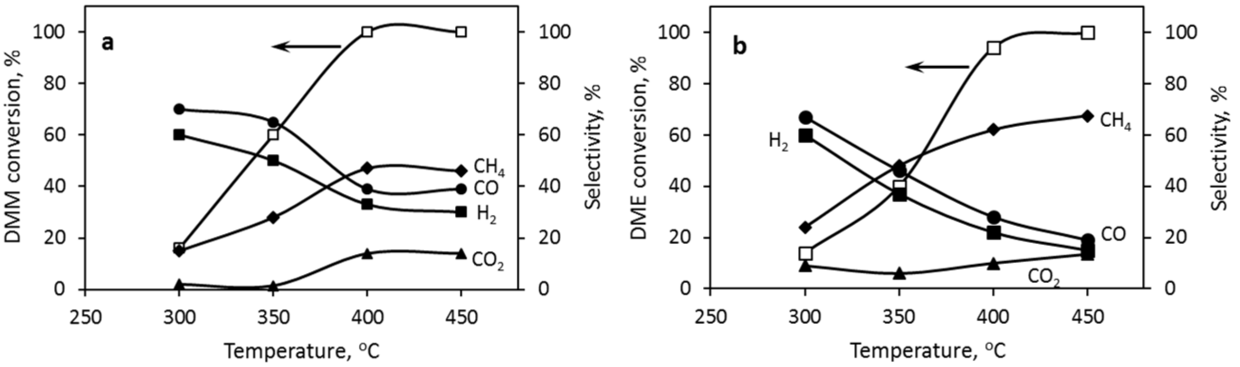

Fig. 3. Effect of temperature on DMM (a) and DME (b) conversion and product selectivity (H_2, CO, CO_2, CH_4) in DMM (a) DME (b) decomposition over Pt/CeO_2-ZrO_2 catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h^{-1}. Inlet composition: $CH_3OCH_2OCH_3$:N_2 = 10:90 vol. % for DMM decomposition and CH_3OCH_3:N_2 = 10:90 vol. % for DME decomposition.

In general, the data in Fig. 3 indicate that the reactions of DMM and DME decomposition on Pt/CeO_2-ZrO_2 are less efficient than FA and methanol decomposition, and even than DMM and DME steam reforming [9, 14]. The most likely explanation to this fact is that the temperature of complete DMM and DME conversion coincides with the temperature of side reaction of CO methanation. The hydrogen-rich gas mixture with high content of methane resulting from DMM and DME decomposition reactions can be used for SOFC feeding but the efficiency of SOFC operation with such a fuel stays behind that of SOFC fuelled by pure hydrogen [22].

In addition, during DMM and DME decomposition experiments (Fig. 3), deactivation of the Pt/CeO_2-ZrO_2 catalyst was observed. The catalyst performance before and after the cycles of reaction temperature increase-decrease was different. Fig. 4 shows the effect of time on-stream on the DMM and DME conversions (a), the H_2 productivity (b) in decomposition of DMM (1) and DME (2) over the Pt/CeO_2-ZrO_2 catalyst. Obviously, the catalyst provided stable operation during 5 h at 350 °C. As Fig. 4 shows, during the first hour on-stream, the Pt/CeO_2-ZrO_2 demonstrated continuous deactivation in both DMM and DME decomposition reactions: DMM conversion decreased from 68 to 55 % and DME conversion - from 39 to 31 % (Fig. 4a); H_2 productivity – from ~1.3 to 1 L/(g_{cat} · h) and from 0.4 to 0.33 L/(g_{cat} · h) (Fig. 4a), respectively, for DMM and DME decomposition. Then the conversions and H_2 productivity remained almost unchanged with time. The TPO studies of the catalysts after DMM and DME decomposition reactions for 5 h revealed carbon formation in the amounts of 0.8 and 0.43 wt. % (to the weight of the catalyst), respectively. Note that the carbon deposition was proportional to the conversion values. In addition, comparative analysis (Fig. 4) shows that Pt/CeO_2-ZrO_2 decomposes DMM more efficiently than DME: DMM conversion almost twice exceeds that of DME, H_2 productivity - 3 times.

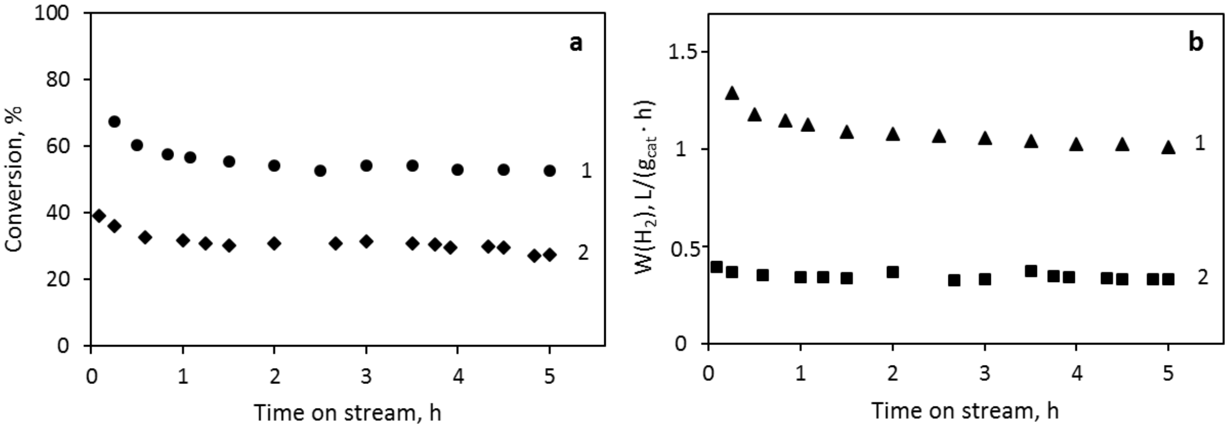

Fig.4. Effect of time-on-stream on conversion (a) and H_2 productivity (b) in DMM (1) and DME (2) decomposition over Pt/CeO_2-ZrO_2 catalyst. Reaction conditions: P = 1 atm; T = 350 °C; GHSV = 10000 h^{-1}. Inlet composition: $CH_3OCH_2OCH_3$:N_2 = 10:90 vol. % for DMM decomposition and CH_3OCH_3:N_2 = 10:90 vol. % for DME decomposition.

Although the Pt/CeO_2-ZrO_2 catalyst was not sufficiently stable during DMM and DME decomposition to hydrogen-rich gas, it seems reasonable to perform further studies in order to improve its activity and stability, and understand in more details the reaction mechanism.

3.4. Comparison of Pt/CeO_2-ZrO_2 performance in FA, methanol, DMM and DME decomposition reactions

Finally, it seems reasonable to summarize the Pt/CeO_2-ZrO_2 performance in FA, methanol, DMM and DME decomposition reactions. Table 1 presents the following data: the temperature of complete conversion of oxygenates into H_2-containing gas; environmental safety of feedstock; main reaction products; favorable fuel cell type.

As shown in Table 1, FA and methanol, despite their corrosiveness and toxicity, decompose with high efficiency into hydrogen-rich gas over the Pt/CeO_2-ZrO_2 catalyst at relatively low temperatures (< 300 °C). Note again that hydrogen-rich gas produced by FA decomposition is suitable for feeding both HT PEMFCs and SOFCs, while that generated by methanol decomposition - only for SOFC feeding.

On the contrary, DMM and DME, being environmentally friendly compounds, demonstrated low hydrogen productivity in decomposition reactions over the Pt/CeO_2-ZrO_2 catalyst. Complete conversion of DMM and DME was achieved at higher temperatures (~400 °C). Moreover, regardless the feedstock type (DMM, DME), the catalyst yielded hydrogen-containing gas with high methane content which can also be used directly to feed SOFCs.

Table 1. Performance of the Pt/CeO_2-ZrO_2 catalyst in FA, methanol, DME and DMM decomposition to hydrogen-rich gas.

Type of fuel	Safety	T, oC	Main products	Favorable fuel cells
Formic acid	Corrosiveness	200-300	H2, CO2	HT PEMFCs* SOFCs**
Methanol	Toxity	250-300	H2, CO	SOFCs
Dimethyl ether	Nontoxic, noncorrosive	400	CH4, H2, CO	SOFCs
Dimethoxymethane	Nontoxic, noncorrosive	400	CH4, H2, CO	SOFCs

* HT PEMFCs – high temperature polymer electrolyte membrane fuel cells;
** SOFCs - solid oxide fuel cells.

Conclusions

Catalytic decomposition of formic acid, methanol, DMM and DME into hydrogen-rich gas was investigated over 1.9 wt.% Pt/CeO_2-ZrO_2 catalyst at 150-450 °C, atmospheric pressure, GHSV = 10000 h^{-1} and feedstock:N_2 = 10:90 (vol.%).

Comparative investigations show that formic acid and methanol decomposition reactions are more efficient than DMM and DME decomposition. The Pt/CeO_2-ZrO_2 catalyst provided complete conversion of formic acid and methanol to hydrogen-rich gas with high H_2 selectivity (> 94 %) at 250-300 °C. Unlike formic acid and methanol, DMM and DME decompose over the

Pt/CeO_2-ZrO_2 catalyst at higher temperatures ~400 °C, so produced hydrogen-rich gas contains significant amount of methane. The Pt/CeO_2-ZrO_2 catalyst was not sufficiently stable in DMM and DME decomposition reactions. Therefore, our further studies are aimed at developing a more active and stable catalyst and probable mechanism of DMM and DME decomposition.

Acknowledgements

This work was supported by the Ministry of Science and Higher Education of the Russian Federation (Project AAAA-A17-117041710088-0).

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КАТАЛИТИЧЕСКОЕ РАЗЛОЖЕНИЕ КИСЛОРОДСОДЕРЖАЩИХ ОРГАНИЧЕСКИХ СОЕДИНЕНИЙ ХИМИИ C_1 В ВОДОРОДСОДЕРЖАЩИЙ ГАЗ

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Аннотация: Исследованы реакции каталитического разложения кислородсодержащих органических соединений химии C_1 в водородсодержащий газ на катализаторе Pt/CeO_2-ZrO_2. Показано, что этот катализатор при атмосферном давлении и температуре до 450 ºC обеспечивает полное разложение муравьиной кислоты, метанола, диметоксиметана и диметилового эфира. Обсуждаются преимущества и недостатки каждой реакции для получения водородсодержащего газа для питания топливных элементов.
Ключевые слова: Каталитическое разложение, оксигенаты, соединения химии C_1, муравьиная кислота, метанол, диметоксиметан и диметиловый эфир, платина, водород, синтез газ.

C_1 OKSİGENTƏRKİBLİ ÜZVİ BİRLƏŞMƏLƏRİN HİDROGENTƏRKİBLİ QAZLARA KATALİTİK PARÇALANMASI

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C_1 oksigentərkibli üzvi birləşmələrin Pt/CeO_2-ZrO_2 katalizatorun iştirakı ilə hidrogentərkibli qazlara katalitik parçalanma reaksiyası tədqiq olunub. Göstəlilib ki, bu katalizator atmosfer təzyiqində və 450 ^{0}C temperaturda qarışqa turşusunun, metanolun, dimetoksimetanın və dimetilefirin tam parçalanmasını təmin edir. Yanacaq elementləri üçün hidrogen qazının alınmasında bu reaksiyaların hər birinin üstünlüyü və çatışmayan cəhətləri müzakirə olunub.
Açar sözlər: katalitik parçalanma**,** qarışqa turşusu, metanol, dimetoksimetan, dimetilefiri, platin, hidrogen.