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## HYDROGEN-RICH GAS PRODUCTION BY CATALYTIC DECOMPOSITION OF OXYGENATED COMPOUNDS OF C<sub>1</sub> CHEMISTRY

S.D. Badmaev<sup>1,2</sup>, A.E. Pinigina<sup>1,2</sup>, V.D. Belyaev<sup>1</sup>, V.A. Sobyenin<sup>1</sup>

<sup>1</sup>*Boreskov Institute of Catalysis,  
Prospekt Lavrentieva, 5, Novosibirsk, 630090 Russia*

<sup>2</sup>*Novosibirsk State University,  
Pirogova Str. 2, Novosibirsk, 630090, Russia  
e-mail: sukhe@catalysis.ru*

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**Abstract:** Catalytic decomposition of oxygenated compounds of C<sub>1</sub> chemistry into hydrogen rich gas was studied over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>1</sub> chemistry, formic acid, methanol, dimethyl ether, dimethoxymethane, platinum, hydrogen, synthesis gas

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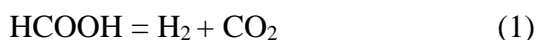
### 1. Introduction

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<sub>1</sub> 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

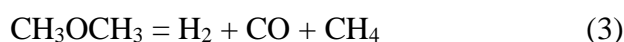
evidence means that no sulfur scrubbing reactor is required. Among the known processes for hydrocarbon catalytic conversion (steam reforming, partial oxidation and decomposition), decomposition is thought to be promising for creating a compact “fuel processor:” this process requires no water or air tanks and respective supply and flow control systems.

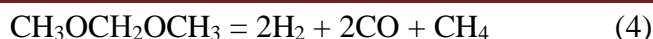
Based on the above facts and taking into account a good performance of Pt-containing catalyst in the decomposition of FA (1) [7], methanol (2) [10], DME (3) [12] and DMM (4) [15], as well as a lower cost of Pt as compared to Pd and Rh, we decided to perform comparative studies of catalytic decomposition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

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



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





### Purpose of the manuscript

The present work reports on the performance of the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> catalyst (1.9 wt % Pt) was prepared by sorption-hydrolytic deposition as described in [17,18]. A solution of H<sub>2</sub>PtCl<sub>4</sub> was mixed with Na<sub>2</sub>CO<sub>3</sub> providing the molar ratio of Na/Cl=1. The obtained mixture was brought into contact with an aqueous suspension of the CeO<sub>2</sub>-ZrO<sub>2</sub> powder (Ecoalliance Ltd. Russia). According to our previous work [18], CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> was ~70 m<sup>2</sup>/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<sub>2</sub>-ZrO<sub>2</sub> catalyst (0.25–0.5 mm) was reduced in situ at 400 °C for 1 h using 10 vol. % H<sub>2</sub>/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<sub>2</sub> for FA decomposition; 10 CH<sub>3</sub>OH and 90 N<sub>2</sub> for methanol decomposition; 10 CH<sub>3</sub>OCH<sub>2</sub>OCH<sub>3</sub> and 90 N<sub>2</sub> for DMM decomposition; 10 CH<sub>3</sub>OCH<sub>3</sub> and 90 N<sub>2</sub> for DME decomposition. Total gas hourly space velocity (GHSV) was 10000 h<sup>-1</sup>.

FA, methanol and DMM were introduced to the reactor by bubbling N<sub>2</sub> through a saturator filled with respective liquid compound. Bronkhorst mass flow controllers fed the DME, N<sub>2</sub> and H<sub>2</sub> (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<sub>i</sub>) and H<sub>2</sub> productivity (W(H<sub>2</sub>)) were calculated using the following equations:

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

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

where  $C_i^0$ ,  $C_{N_2}^0$  are the inlet concentrations (vol.%) of oxygenate (FA, methanol, DME and DMM) and N<sub>2</sub>;

$C_i$ ,  $C_{N_2}$ ,  $C_{H_2}$  are the outlet concentrations (vol.%) of unconverted oxygenate, N<sub>2</sub>, H<sub>2</sub>.

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

The H<sub>2</sub> selectivity (S(H<sub>2</sub>)) for decomposition reactions was calculated as total moles of H<sub>2</sub> actually produced through division by the moles of H<sub>2</sub> 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 (Netzsch, Germany). The feed gas, 6 vol.% O<sub>2</sub>/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<sub>2</sub> concentration was monitored on-line by a QMS-200 mass-spectrometer (Stanford Research Systems, USA). The amount of the coke

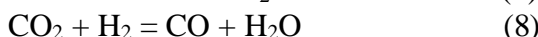
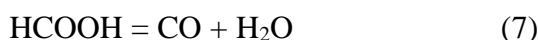
deposited on the catalyst was determined from that of CO<sub>2</sub> released in the TPO runs.

### 3. Results and discussion

#### 3.1. Catalytic decomposition of formic acid

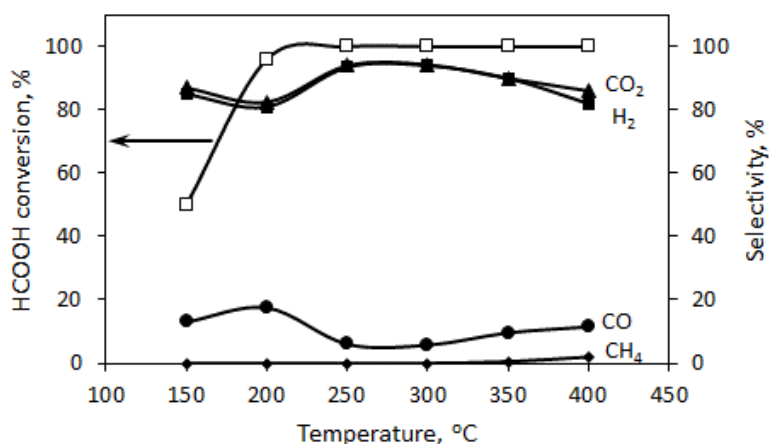
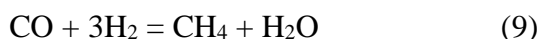
Fig. 1 shows the effect of temperature on FA conversion and product selectivity (H<sub>2</sub>, CO<sub>2</sub>, CO and CH<sub>4</sub>) in FA decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. As the reaction temperature increased from 150 to 400 °C, the FA conversion increased and reached ~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<sub>2</sub> and CO<sub>2</sub> 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:



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]:



**Fig.1.** Effect of temperature on formic acid conversion and product selectivity (H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>) in FA decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h<sup>-1</sup>. Inlet composition: HCOOH:N<sub>2</sub> = 10:90 vol. %.

In general, the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst demonstrates good performance for FA decomposition into hydrogen-rich gas. It provides 100% FA conversion with high (~94 %) H<sub>2</sub> and CO<sub>2</sub> selectivity at 250-300 °C. With consideration of the results obtained, we calculated the hydrogen-rich gas composition (excluding N<sub>2</sub>) after the FA decomposition at

250-300 °C (vol. %): 47 H<sub>2</sub>; 47 CO<sub>2</sub>; 3 H<sub>2</sub>O 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<sub>2</sub>-ZrO<sub>2</sub> catalyst with the predominant formation of H<sub>2</sub> and CO. Fig. 2 shows temperature dependencies of methanol conversion and the selectivity of the main reaction products (H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>) in methanol decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub>

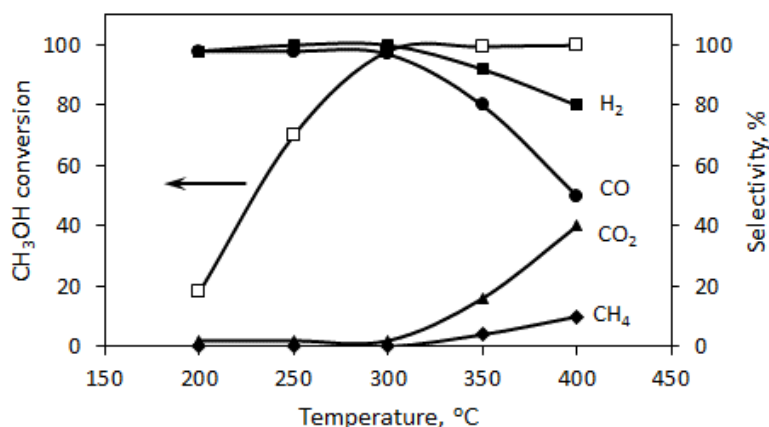
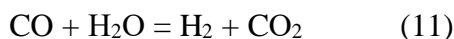
catalyst. At 200 °C, methanol conversion was ~20% which increased as temperature increased to reach ~100 % at 300 °C. H<sub>2</sub> and CO are main reaction products at 200–300 °C; their selectivity was ~100 % suggesting that the methanol decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> proceeds according to reaction (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<sub>2</sub> and CO selectivity decreased,

whereas the CH<sub>4</sub> and CO<sub>2</sub> selectivity increased up to 10 and 40 %, respectively. The formation of CH<sub>4</sub>, CO<sub>2</sub> and H<sub>2</sub>O (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):



**Fig. 2.** Effect of temperature on methanol conversion and product selectivity (H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>) in methanol decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h<sup>-1</sup>. Inlet composition: CH<sub>3</sub>OH:N<sub>2</sub> = 10:90 vol. %.

Thus, Fig. 2 data show that Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> catalyst is quite promising for efficient production of synthesis gas for SOFC feeding applications.

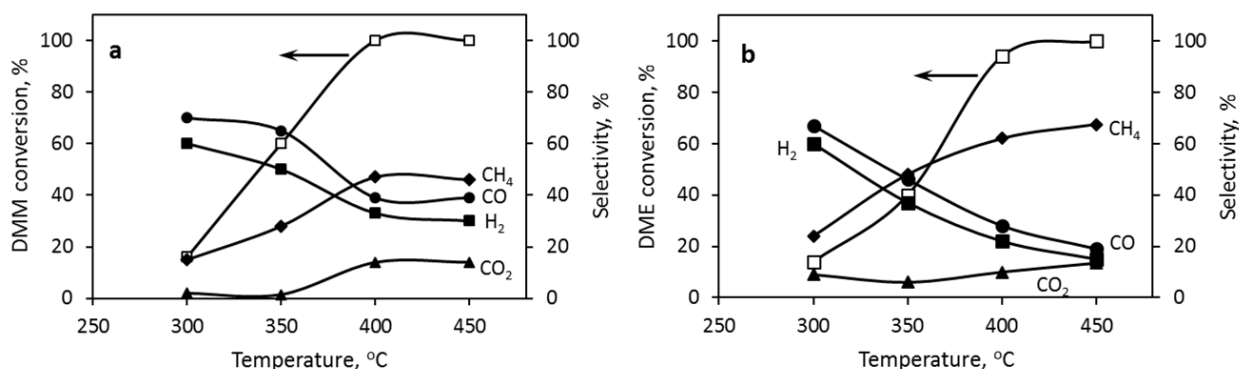
### 3.3. Catalytic decomposition of DMM and DME

Fig. 3 illustrates the effect of temperature on the conversion and product selectivity in DMM and DME decomposition reactions over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. As is seen in Fig. 3, the DMM and DME conversions over Pt/CeO<sub>2</sub>-

ZrO<sub>2</sub> 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<sub>2</sub>, CO, CH<sub>4</sub> and CO<sub>2</sub> 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<sub>2</sub> 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<sub>2</sub> and CO selectivity decreased from 60-70% to 20-40% with simultaneous increase in CH<sub>4</sub> formation up to 50-70 %. Note that the CO<sub>2</sub> 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<sub>4</sub> and CO<sub>2</sub> by, for example, side-reactions (9) and (11).



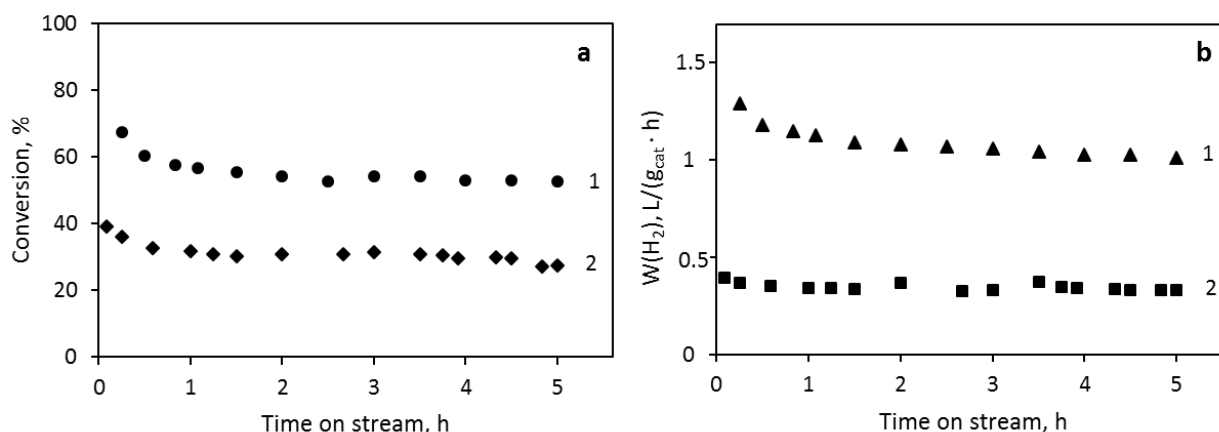
**Fig. 3.** Effect of temperature on DMM (a) and DME (b) conversion and product selectivity (H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>) in DMM (a) DME (b) decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. Reaction conditions: P = 1 atm; GHSV = 10000 h<sup>-1</sup>. Inlet composition: CH<sub>3</sub>OCH<sub>2</sub>OCH<sub>3</sub>:N<sub>2</sub> = 10:90 vol. % for DMM decomposition and CH<sub>3</sub>OCH<sub>3</sub>:N<sub>2</sub> = 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<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub> productivity (b) in decomposition of DMM (1) and DME (2) over the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub> productivity - from ~1.3 to 1 L/(g<sub>cat</sub> · h) and from 0.4 to 0.33 L/(g<sub>cat</sub> · h) (Fig. 4a), respectively, for DMM and DME decomposition. Then the conversions and H<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> decomposes DMM almost twice exceeds that of DME, H<sub>2</sub> more efficiently than DME: DMM conversion productivity - 3 times.



**Fig.4.** Effect of time-on-stream on conversion (a) and H<sub>2</sub> productivity (b) in DMM (1) and DME (2) decomposition over Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst. Reaction conditions: P = 1 atm; T = 350 °C; GHSV = 10000 h<sup>-1</sup>. Inlet composition: CH<sub>3</sub>OCH<sub>2</sub>OCH<sub>3</sub>:N<sub>2</sub> = 10:90 vol. % for DMM decomposition and CH<sub>3</sub>OCH<sub>3</sub>:N<sub>2</sub> = 10:90 vol. % for DME decomposition.

Although the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> performance in FA, methanol, DMM and DME decomposition reactions

Finally, it seems reasonable to summarize the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> performance in FA, methanol, DMM and DME decomposition reactions. Table 1 presents the following data: the temperature of complete conversion of oxygenates into H<sub>2</sub>-containing gas; environmental safety of feedstock; main reaction products; favorable fuel cell type.

**Table 1.** Performance of the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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	H <sub>2</sub> , CO <sub>2</sub>	HT PEMFCs* SOFCs**
Methanol	Toxity	250-300	H <sub>2</sub> , CO	SOFCs
Dimethyl ether	Nontoxic, noncorrosive	400	CH <sub>4</sub> , H <sub>2</sub> , CO	SOFCs
Dimethoxymethane	Nontoxic, noncorrosive	400	CH <sub>4</sub> , H <sub>2</sub> , CO	SOFCs

\* HT PEMFCs – high temperature polymer electrolyte membrane fuel cells;

\*\* SOFCs - solid oxide fuel cells.

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<sub>2</sub>-ZrO<sub>2</sub> 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<sub>2</sub>-ZrO<sub>2</sub> 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.

### Conclusions

Catalytic decomposition of formic acid, methanol, DMM and DME into hydrogen-rich gas was investigated over 1.9 wt.% Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst at 150-450 °C, atmospheric pressure, GHSV = 10000 h<sup>-1</sup> and feedstock:N<sub>2</sub> = 10:90 (vol.%).

Comparative investigations show that formic acid and methanol decomposition reactions are more efficient than DMM and DME decomposition. The Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst provided complete conversion of formic acid and methanol to hydrogen-rich gas with

high H<sub>2</sub> selectivity (> 94 %) at 250-300 °C. Unlike formic acid and methanol, DMM and DME decompose over the Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> catalyst at higher temperatures ~400 °C, so produced hydrogen-rich gas contains significant amount of methane. The Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> 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

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**КАТАЛИТИЧЕСКОЕ РАЗЛОЖЕНИЕ КИСЛОРОДСОДЕРЖАЩИХ ОРГАНИЧЕСКИХ СОЕДИНЕНИЙ ХИМИИ C<sub>1</sub> В ВОДОРОДСОДЕРЖАЩИЙ ГАЗ****С.Д. Бадмаев<sup>1,2</sup>, А.Е. Пинигина<sup>1,2</sup>, В.Д. Беляев<sup>1</sup>, В.А. Собянин<sup>1</sup>**<sup>1</sup> *Институт катализа им. Г.К. Борескова Сибирского отделения РАН,  
пр. Лаврентьева 5, г. Новосибирск, 630090 Россия*<sup>2</sup> *Новосибирский государственный университет,  
ул. Пирогова 2, г. Новосибирск, 630090 Россия  
e-mail:sukhe@catalysis.ru*

**Аннотация:** Исследованы реакции каталитического разложения кислородсодержащих органических соединений химии C<sub>1</sub> в водородсодержащий газ на катализаторе Pt/CeO<sub>2</sub>-ZrO<sub>2</sub>. Показано, что этот катализатор при атмосферном давлении и температуре до 450 °С 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<sub>1</sub>, муравьиная кислота, метанол, диметоксиметан и диметиловый эфир, платина, водород, синтез газ.

**C<sub>1</sub> OKSİGENTƏRKİBLİ ÜZVİ BİRLƏŞMƏLƏRİN HİDROGENTƏRKİBLİ QAZLARA KATALİTİK PARÇALANMASI****S.D. Badmayev<sup>1,2</sup>, A.E. Pinigina<sup>1,2</sup>, V.D. Belyayev<sup>1</sup>, V.A. Sobyenin<sup>1</sup>**<sup>1</sup> *REA Sibir şöbəsi, G.K. Boreskov adına Kataliz İnstitutu.  
Lavrentieva prospekti 5, Novosibirsk, 630090 Rusiya*<sup>2</sup> *Novosibirsk Dövlət Universiteti,  
Pirogova küç. 2, Novosibirsk, 630090 Rusiya  
e-mail:sukhe@catalysis.ru*

C<sub>1</sub> oksigentərkibli üzvi birləşmələrin Pt/CeO<sub>2</sub>-ZrO<sub>2</sub> katalizatorun iştirakı ilə hidrogentərkibli qazlara katalitik parçalanma reaksiyası tədqiq olunub. Göstərilib ki, bu katalizator atmosfer təzyiqində və 450 °С 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.