



Stability and Reactivity of a Polyoxymethylene Dimethyl Ether over Typical Catalysts for Diesel Emission Control

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Abstract

Polyoxymethylene dimethyl ethers (OME) produced from methanol are considered as potential substitutes of Diesel fuel. Emissions of formaldehyde and other components have been observed, particularly under cold-start conditions in engine test-bench experiments with OME fuel. In this study, the reactivity of OME₃ (CH₃O(CH₂O)₃CH₃) and its decomposition products was studied in the temperature range 80–450 °C in a model gas test bench over V₂O₅/WO₃/TiO₂ and Cu-CHA SCR catalysts, a platinum-coated V₂O₅/WO₃/TiO₂ ammonia slip catalyst (ASC) and two diesel oxidation catalysts (DOC), based on platinum and platinum-palladium. Already at 80 °C, OME₃ was largely hydrolyzed to methanol and formaldehyde over all catalysts. At temperatures above 150 °C, V₂O₅/WO₃/TiO₂ oxidized methanol and formaldehyde to CO via formic acid as intermediate. The platinum ASC showed a similar behavior but oxidized the decomposition products to CO₂. Whereas Cu-CHA hydrolyzed OME₃ quantitatively to methanol and formaldehyde, it did not show oxidation activity in the studied temperature range. The data indicate that the release of significant amounts of OME from a catalytic converter can be virtually ruled out under cold start conditions, but also that low temperature hydrolysis produces formaldehyde and methanol emissions.

Keywords Exhaust gas catalysts · Polyoxymethylene ethers · Methanol · Formaldehyde · Formic acid · Selective catalytic reduction · Diesel oxidation catalysts · Ammonia slip catalysts · Oxidation · Hydrolysis

1 Introduction

Polyoxymethylene dimethyl ether (OME) is an emerging synthetic oxygenated fuel with potential use as CO₂-neutral and sustainable Diesel substitute or additive. Due to its high oxygen content, less hydrogen and thus also less energy is required for its production compared to Fischer-Tropsch fuels [1]. Since OME is classified as a second generation synfuel, the environmental impact of its production is relatively low [2]. OME with three to five oxymethylene units (OME_{3–5}) exhibits a shorter ignition delay than Diesel fuel (cetane number ≥ 70), vaporizes easily and allows high EGR

rates with low exhaust emissions. Due to these advantageous properties, OME_{3–5} has been certified as fuel according to the DIN 51,699 standard, which is a pre-requisite for its market introduction.

Interest in OME has been growing rapidly since 2010 [3, 4]. The majority of OME publications deal with synthesis and motor applications, which have been summarized in a number of review papers [1, 4–11]. In contrast, not much is known about the chemical properties of OME_{3–5}. Only the thermal and oxidative stability of OME fuel has been studied in more detail since this data is required for the standardization of OME_{3–5} [12]. OME₃ (CH₃O(CH₂O)₃CH₃) is stable in water but hydrolysis takes place in the presence of acids [13]. A facile hydrolysis reaction was found at 60 and 90 °C in a reactor filled with the acidic ion-exchange resin Amberlite, forming methanol and formaldehyde [14]:

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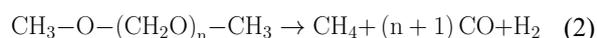
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This is an equilibrium reaction, which allows the simple large-scale synthesis of OME, if water is removed [15]. An excess of water, on the other hand, results in complete hydrolysis with the formation of methanediol.

The fulfillment of extremely strict emission limits that are planned for Diesel engines in the future is possible by replacing Diesel fuel based on hydrocarbons with C-C bonds by OME₃₋₅ with C-O-C bonds, thus avoiding the typical NO_x-soot tradeoff. However, a NO_x-methane tradeoff was found for OME fuel that limits engine internal measures for NO_x reduction [16]. In the case of oxygen deficit, the formation of methane is likely due to pyrolysis of OME via formaldehyde as intermediate product:



A remaining problem is the reduction of cold start emissions. Although OME₃₋₅ burns completely during normal engine operation, there is a risk that unburned OME and its decomposition products will be released to the exhaust gas during cold start or low load operation. In Diesel engines, electric catalyst heating and heating by fuel dosing have been applied as countermeasures. However, Diesel fuel dosing at low exhaust temperatures in order to heat up the following after-treatment system by initiating exothermic reactions inside of the DOC is limited by the slip of hydrocarbon emissions. This measure is realized in the engine by late post injection of Diesel fuel in the cylinder and results in increasing raw engine emissions of hydrocarbons. In case of OME, a reciprocal proportionality between injected fuel amount and non-oxidized OME emissions downstream of the DOC were observed, even at low exhaust temperatures of 180 °C. However, the OME injected for catalyst heating causes formaldehyde and methane emissions [17], which have to be avoided. The interaction of OME with DOC and SCR catalysts in Diesel engines is largely unknown. Recently, first results of the light-off behavior of OMEs over a commercial oxidation showed that this class of compounds produced formaldehyde as decomposition product already at 100 °C that is oxidized by the oxidation catalyst at higher temperatures [18].

The observed decomposition products of OME at low exhaust gas temperatures necessitates the evaluation of the interaction of OME with currently used catalyst technologies (DOC and SCR) in Diesel engines to derive guidelines for the development of suitable exhaust gas systems in OME-burning engines. To this end, we have measured in an exploratory study the hydrolysis and oxidation of OME₃ and its decomposition products on current catalyst technologies in a model gas test rig in the temperature range

Table 1 Catalyst samples and tested space velocities. SCR = selective catalytic reduction, ASC = ammonia slip catalyst, DOC = Diesel oxidation catalyst

Catalyst designation	Composition	Type	Cell density [h ⁻¹] [cpsi]	GHSV
VWT	2 wt% V ₂ O ₅ /10 wt% WO ₃ /TiO ₂	Extrudate	300	45,000
Pt/VWT	5 g Pt/ft ³ /2 wt% V ₂ O ₅ /10 wt% WO ₃ /TiO ₂	Extrudate	300	45,000
Cu-CHA	2.6 wt% Cu-CHA	Coated cordierite substrate	400	45,000
DOC A	90 g Pt/ft ³	Coated cordierite substrate	400	65,000
DOC B	20 g (Pt+Pd)/ft ³ + zeolite	Coated cordierite substrate	400	65,000

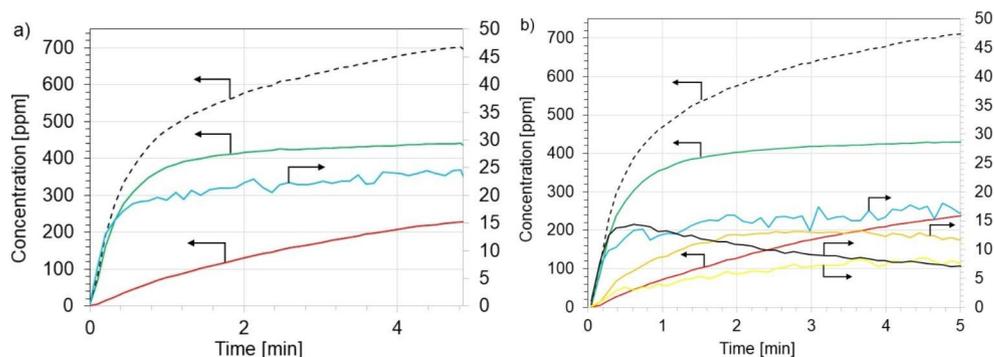
80–450 °C with the aim to evaluate the stability of OME₃ at low temperatures and its reactivity.

2 Experimental

2.1 Model gas test rig and Catalysts

The measurements to study the behavior of OME over various exhaust gas catalysts were conducted with a model gas test rig (Fig. S1). The feed gas was obtained by mixing pure gases using mass flow controllers. Water was generated by oxidation of hydrogen on a Pt/Al₂O₃ catalyst thus ensuring a pulsation-free production. OME₃ was added to the flow to the reactor by passing nitrogen through an OME₃-containing wash bottle at room temperature. All sample lines and the gas pump between the setup and the FTIR spectrometer were heated to 80 °C, which is sufficiently high to avoid condensation but low enough to limit the blank conversion of OME to formaldehyde to a few percent. The catalyst samples were tested in a quartz reactor with an inner diameter of 30 mm. The reactor consisted of several heating zones, including a preheating zone filled with ceramic beads that guaranteed perfect temperature mixing of the model gas across the reactor cross-section. The temperature of the individual zones was controlled by thermocouples, which were positioned in the center of the gas flow. One thermocouple upstream and one downstream of the catalyst module were used to control the temperature of the catalyst. The catalyst module to be tested was placed in a round catalyst mount (Ø 28 mm) with a rectangular opening (15 × 20 mm) to accommodate the module. The catalyst modules (13 × 17 × 38–50 mm) were sealed in the catalyst mount with a ceramic fiber mat, and the mount itself was inserted into the reactor wrapped in another ceramic mat.

Fig. 1 Isothermal decomposition of OME₃ in wet feed over (a) VWT and (b) Pt/VWT at 80 °C. Formaldehyde (green colour —), methanol (red colour —), OME₃ (light blue colour —), formic acid (orange colour —), CO₂ (black colour —), methyl formate (yellow colour —), sum of C1 carbon components (- - -)



The following commercial catalysts were tested for the OME hydrolysis and oxidation activity (Table 1). A commercial extruded V₂O₅/WO₃/TiO₂ SCR catalyst with a cell density of 300 cpsi (VWT), a platinum-loaded V₂O₅/WO₃/TiO₂ SCR catalyst as ammonia slip catalyst (Pt/VWT), a Cu-CHA catalyst (Cu-CHA) and two commercial Diesel oxidation catalysts (DOC A and B) with very high platinum and medium platinum + palladium loading, respectively. DOC B contained a zeolite for adsorption of hydrocarbons during cold start of the engine.

The catalytic tests were performed in a feed containing ca. 800 ppm_{C1} (ppm_{C1} = concentration in C1 equivalents) OME₃ (from ASG Analytik-Service AG), 10 vol% O₂ and 5 vol% H₂O in nitrogen from 80 to 450 °C at a rate of 10 °C·min⁻¹. The temperature ramps also allowed to detect adsorption and desorption phenomena on the catalyst surface by calculating the carbon balance of the desorbed products observed by online gas phase FTIR spectroscopy.

2.2 FTIR gas Analysis

The gas downstream of the catalyst was sampled using a heated gas pump (KNF; Teflon membranes, 170 °C) and fed with heated steel lines to a Thermo-Nicolet Nexus FTIR spectrometer equipped with a heated 2 m gas cell (180 °C). The gaseous components were quantified using the Omnic/Quantpad software. An existing quantification method for the measurement of formaldehyde and its reaction products was extended with OME₃. Fig. S2 shows the FTIR absorption spectrum of 400 ppm_{C1} OME₃. The absorption bands from 900 cm⁻¹ to 1300 cm⁻¹ were used for the quantification and calibration. Measured components and their detection limits: CO (0.2 ppm), CO₂ (0.5 ppm), OME₃ (1–2 ppm_{C1}), formaldehyde (0.5 ppm), methanol (2 ppm), formic acid (1–2 ppm), methyl formate (1 ppm). The cross-sensitivity of OME₃ to formic acid could not be completely eliminated at high OME₃ concentrations. Therefore, the detection limit of formic acid applies only to OME₃ concentrations below 100 ppm. Higher concentrations, which occurred only rarely during the measurements, caused negative formic acid concentrations of a few ppm.

3 Results and Discussions

First isothermal experiments with a feed containing 1000 ppm_{C1} OME₃, oxygen and water through the empty reactor heated at 80 °C yielded 15–20% OME₃ conversion to formaldehyde, demonstrating the instability of OME already at moderate temperatures. This instability became more apparent when OME₃ was dosed over different catalysts at 80 °C. OME₃ was largely converted (98%) to formaldehyde and methanol, from which it is produced, over both VWT and Pt-VWT (Fig. 1) in wet feed. The slowly increasing methanol concentration at the reactor outlet indicates that methanol was adsorbed on the catalyst surface and equilibrium was not completely reached before the experiment was stopped. This conclusion is confirmed by the parallel increase of the carbon balance. Whereas formic acid, methyl formate and CO₂ were virtually absent over VWT, small amounts were formed over Pt-VWT due to oxidation of formaldehyde and the condensation reaction to methyl formate. In dry feed, OME₃ conversion was lower over VWT but still ca. 74% at 80 °C (not shown). On Cu-CHA, DOC A and DOC B, the isothermal conversion was lower than with VWT and Pt-VWT but still very high (Cu-CHA: 95%, DOC A: 94%, DOC B: 95%, not shown), suggesting that all catalysts efficiently hydrolyzed OME₃ already at 80 °C.

Figure 2 shows the reaction products during the conversion of OME₃ on the different catalysts during temperature-programmed reaction from 80 to 450 °C. OME₃ was almost quantitatively converted to formaldehyde and methanol up to 150 °C over VWT. The small concentrations of OME₃ observed at 80–100 °C were virtually completely converted above 200 °C. Methanol was increasingly oxidized to formaldehyde above 150 °C. In parallel, formaldehyde started to be oxidized to formic acid that reacted with methanol to traces of methyl formate. The increasing levels of CO above 175 °C were associated with the decomposition of formic acid. Around 200 °C, the formaldehyde formation exhibited a maximum of ca. 550 ppm that coincided with the maximum of the carbon balance (Fig. 3). This is indicative of the presence of adsorbed species, which were released

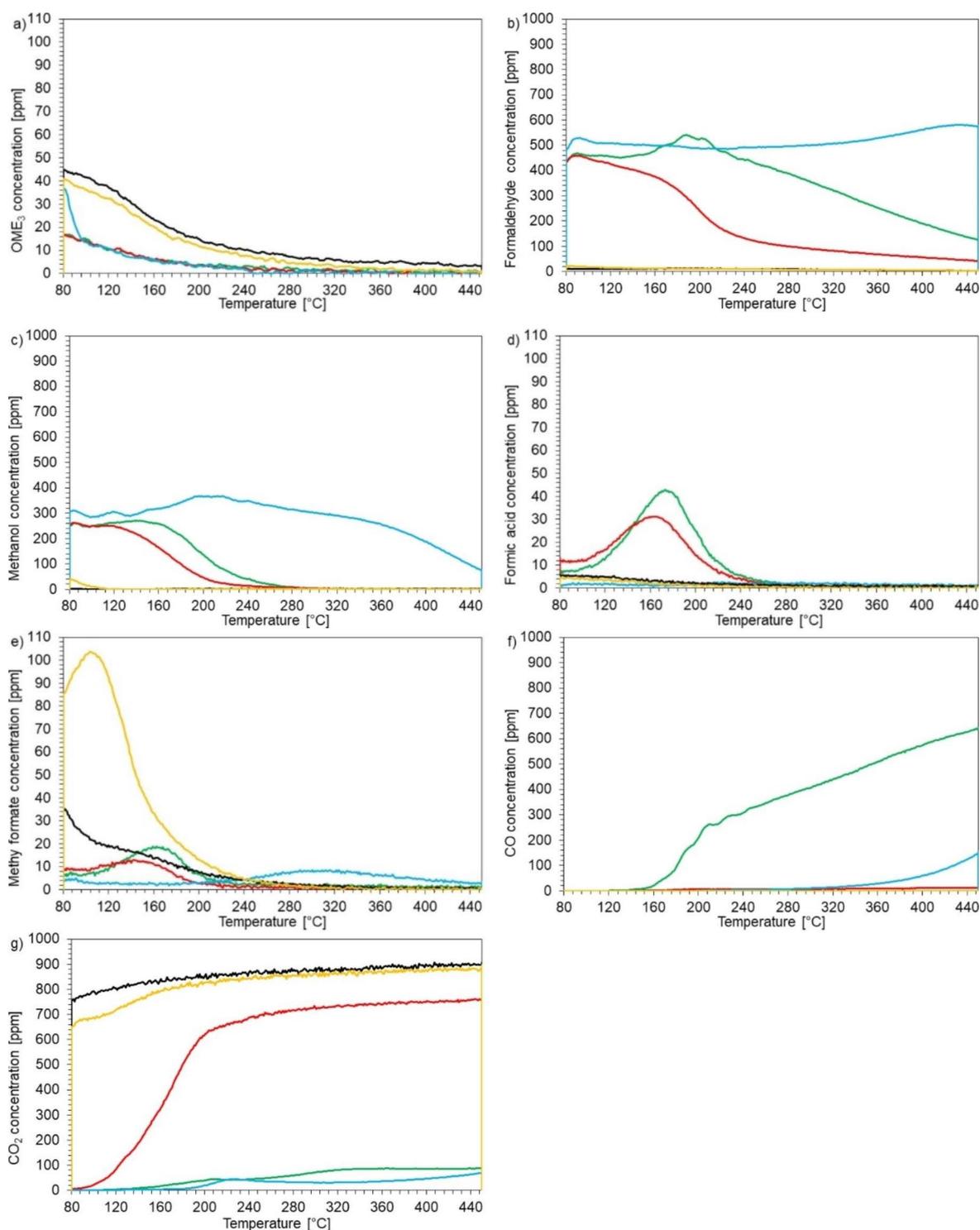


Fig. 2 Emissions of (a) OME₃, (b) formaldehyde, (c) methanol, (d) formic acid, (e) methyl formate, (f) CO and (g) CO₂ during temperature-programmed reaction from 80 to 450 °C upon dosage of 800 ppm_{C1} OME₃, 10 vol% O₂ and 5 vol% H₂O in nitrogen at GHSV = 45,000 h⁻¹

over VWT (green colour —), Pt/VWT (red colour—), Cu-CHA (light blue colour—) and at GHSV = 65,000 h⁻¹ over DOC A (black colour —) and DOC B (yellow colour—)

predominantly as formaldehyde in this temperature range. Above 250 °C, formaldehyde, CO and CO₂ were the dominant products. Although the formaldehyde concentration

decreased steadily up to 450 °C, still 130 ppm were detected at this temperature.

The influence of water on the decomposition of OME_3 and its reaction products was tested in a control experiment in dry feed gas (Fig. S3). In this case, the conversion of OME_3 at 80 °C was significantly lower resulting in OME_3 emissions of about 200 ppm, which is about 10 times higher than in the presence of water. With increasing temperature, however, the OME_3 conversion increased rapidly and reached the same low OME_3 concentrations after catalyst as in the presence of water. It is noteworthy that at the start of the temperature ramp at 80 °C, the carbon balance was significantly lower (68%) than in the presence of water (85%), indicating that a larger fraction of reaction products and intermediates were adsorbed at low temperatures, which desorbed in the range between 150 and 250 °C (max. carbon balance 160%, Fig. 3). Above 250 °C, the same reaction prevailed in the absence and presence of water, i.e. increasing oxidation of formaldehyde to CO. Due to the missing inhibitory effect of surface hydroxyl groups, the oxidation activity of the catalyst was somewhat higher in the dry feed gas and thus the formaldehyde concentrations of 90 ppm at 450 °C were slightly lower.

The same catalyst modified by platinum to function as ammonia slip catalyst (ASC) was tested next. Below 150 °C, virtually no differences were found between Pt/VWT and VWT. The OME_3 conversion and the evolution of the formaldehyde, methanol, formic acid and methyl formate were similar in this temperature range. The main difference was the formation of CO_2 , which already started at low temperatures. Above 200 °C, CO_2 became the main product of OME_3 conversion and oxidation. Above 150 °C, the oxidation activity of platinum in Pt-VWT increased sharply, so that at 200 and 300 °C much lower formaldehyde concentrations (121 ppm) were found downstream of the catalyst compared to VWT (427 ppm). However, the decrease of the formaldehyde emissions from this lower value continued at a much lower rate.

When using the Cu-CHA catalyst, mainly formaldehyde and methanol were observed in the ratio 3:2 at temperatures up to 170 °C in accordance with a stoichiometric decomposition of OME_3 . This demonstrates the strong activity of the acidic Cu-CHA catalyst for OME_3 hydrolysis. During the temperature ramp, the concentration of formaldehyde changed only slightly up to 300 °C, as did that of methanol, which went through a maximum between 200 and 250 °C. In parallel, there was a slight increase in the carbon balance (Fig. 3), which can be explained by desorption of small amounts of previously adsorbed species. Cu-CHA showed a significantly lower oxidation activity than VWT, noticeable from the appreciable amounts of ca. 80 ppm methanol at 450 °C. Although methanol could be oxidized to formaldehyde, apparently its oxidation potential was too small for the oxidation of formaldehyde to CO and CO_2 leading

to the significant increase in formaldehyde concentrations between 250 and 450 °C. The subsequent oxidation to CO and CO_2 was on a very low level with 70 ppm CO_2 and 150 ppm CO at 450 °C. Despite its rather low activity towards OME_3 oxidation, Cu-CHA catalyzed very efficiently OME_3 hydrolysis. At 80 °C, 38 ppm OME_3 were found downstream of the catalyst, which decreased to the detection limit of 1 ppm above 250 °C.

When comparing the results of the SCR-type catalysts, it is clearly discernible that a large fraction of OME_3 was already hydrolyzed at 80 °C. Whereas for VWT and Pt/VWT almost 20 ppm OME_3 were found, 36 ppm were found over Cu-CHA. The behavior of all catalysts was very similar at higher temperatures. Cu-CHA showed complete OME_3 conversion already at 250 °C.

As expected from the high platinum content of DOC A, CO_2 was the main reaction product of the OME_3 conversion already at 80 °C. However, the conversion was significantly lower than that over VWT and Pt/VWT. Up to 100 °C, more than 40 ppm of unreacted OME_3 were found, more than twice as much as after the VWT-containing catalysts. The OME_3 concentrations also decreased only slowly with increasing temperature and were still measurable at 400 and 450 °C (3–5 ppm). Similarly, formaldehyde emissions were only 10 to 12 ppm between 80 and 250 °C, but decreased very slowly with increasing temperature, so that about 3–5 ppm formaldehyde were still found at 400 and 450 °C. The virtually constant carbon balance demonstrates that adsorption and desorption of reaction products is negligible on DOC A in the studied temperature range (Fig. 3).

Over DOC B, which contains lower amounts of platinum and palladium, the OME_3 conversion was similar to the highly loaded DOC A. The OME_3 concentrations downstream of the catalyst decreased during the heating ramp from ca. 40 ppm at 80 °C to ca. 10 ppm at 200 °C, but remained at a few ppm with further increasing temperature. Temperatures above 400 °C were required to quantitatively convert OME_3 . The only product of the oxidation was CO_2 that was accompanied by a few ppm formaldehyde. It is remarkable that this catalyst produced high concentrations of methyl formate in the lower temperature range (100 ppm at 100 °C).

The hydrolysis activity of the two DOC catalysts can be compared best at 80 °C, where both samples showed significantly higher OME_3 emissions of ca. 40 ppm than VWT, which were also slightly higher than those of Cu-CHA. In addition, they showed a much slower decrease in the emissions with increasing temperature. A remarkable feature of the very strong oxidation catalyst DOC A were the remaining 3–5 ppm OME_3 even at 400 and 450 °C. Such persistent OME_3 emissions were not observed over DOC B, which

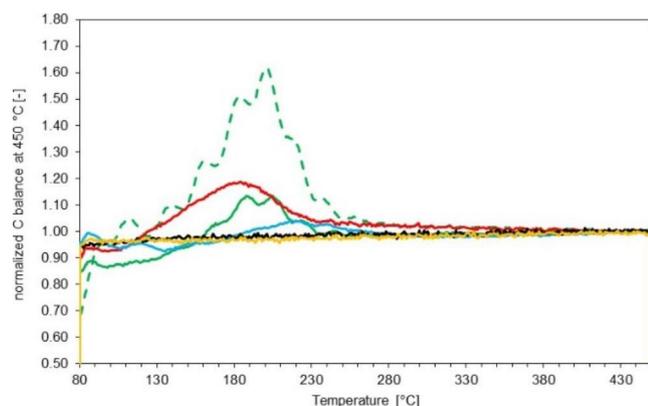


Fig. 3 Normalized carbon balances for all catalysts. VWT (green colour —), Pt/VWT (red colour —), Cu-CHA (light blue —), DOC A (black colour —) and DOC B (yellow colour —)

Table 2 Normalized amounts of desorbed carbon during the catalytic tests. These amounts were used to estimate the amounts of adsorbates per liter of catalyst, assuming that the adsorbates are composed of formaldehyde

	Water concentration	Desorbed amounts		
	%	mmol C	mg _{CH₂O}	mg _{CH₂O} /L
VWT	0	0.90	27	3000
VWT	5	0.13	4	440
Pt/VWT	5	0.36	11	1200
Cu-CHA	5	0.05	1.4	134
DOC A	5	<0.01	<0.3	<50
DOC B	5	<0.01	<0.3	<50

contained also zeolites that were most probably responsible for the hydrolysis of the OME₃ under these conditions.

Regarding oxidation reactions, the two precious metal-containing DOCs showed formaldehyde concentrations in the range of only 10–20 ppm at 80 °C, but CO₂ as main product instead. The formaldehyde slip remained at a level of 10 ppm up to about 250 °C and decreased only to values of 3–5 ppm even at 450 °C. Consistent with the very high oxidation activity of these catalysts, one would expect complete formaldehyde conversion at lower temperatures. This can be only explained by the decomposition of OME₃ to formaldehyde and methanol also in the rear part of the catalyst, where the residence time was insufficient for complete oxidation of formaldehyde to CO₂.

The carbon balances in Fig. 3 allow to draw conclusions about the adsorption and desorption of OME₃ and its reaction products. Since mainly formaldehyde was desorbed in the temperature-programmed reaction experiments, the amount of deposits per liter of catalyst were calculated as formaldehyde (mg_{CH₂O}/L) (Table 2). The largest amount of desorbed carbon species (0.90 mmol) was found in the experiment with VWT in the absence of water. Desorption started at about 125 °C and reached its maximum of

160% relative carbon balance at 200 °C compared to the amount expected from the constant dosage of 800 ppm_{C1} OME₃ at 200 °C. The desorbed amount decreased rapidly at higher temperatures and desorption was virtually complete at 250 °C. With water in the feed, the desorbed amount of carbon from this catalyst was only 0.13 mmol, i.e. about 7 times less. Cu-CHA showed only a small desorption peak in the region of 225 °C with 0.05 mmol carbon. Three times more (0.36 mmol) were desorbed from Pt/VWT compared to VWT. A possible explanation for this observation is the only partial oxidation of methanol and formaldehyde to formate/formic acid on platinum at low temperatures (< 125 °C). Due to the strong surface adsorption of formate species, this intermediate remained predominantly adsorbed on the catalyst and was increasingly oxidized to CO₂ only above about 125°. The two DOC did not show any desorption peak. It is reasonable to assume that the primary decomposition products of OME, formaldehyde and methanol, could already be oxidized to CO₂ at 80 °C. The formation of methyl formate on these catalysts implies that formic acid is likely an intermediate in the oxidation of OME as well.

4 Conclusion

The results of our study demonstrate that after cold start of the engine, OME can already be largely converted at 80 °C on all tested catalysts, i.e. VWT and Cu-CHA SCR catalysts, the ASC and the two platinum-based DOC. The release of appreciable amounts of OME, which can adsorb and condense on the surface of after-treatment catalysts after cold start of the engine, can therefore be virtually ruled out. However, OME first hydrolyses to its building blocks formaldehyde and methanol, which could be emitted in the exhaust gas. Catalysts with redox-active components were able to oxidize methanol and formaldehyde further. Whereas VWT SCR catalyst formed CO via formic acid as intermediate at temperature above 150 °C and the platinum-coated VWT catalyst (used as ACS) produced CO₂ instead, the two DOC were highly active for oxidation to CO₂ already at 80 °C. This means that if a DOC and SCR catalyst are combined in an exhaust after-treatment system, it can be expected that the major fraction of OME will be completely oxidized to CO₂ on the DOC after cold start even at low temperatures and the remaining OME traces should be adsorbed and completely converted on the SCR catalyst. Further studies on combined catalyst systems are required to confirm this conclusion.

The use of only an SCR catalyst could be more critical. The experiments with Cu-CHA showed that it was very active in hydrolyzing OME, but was virtually unable to oxidize the formed reaction products to CO₂. The OME residues

and partial decomposition products adsorbed and condensed during a cold start on the surface of Cu-CHA SCR catalyst will leave the after-treatment system as methanol and formaldehyde. VWT-based catalysts are slightly better suited for this purpose, since they can store these products somewhat better during the cold start phase and exhibit oxidation properties to at least partially convert them to CO and CO₂.

One option to increase the efficiency of after-treatment systems is to inject fuel into the exhaust system to raise the exhaust gas temperature. Both DOC A and B tested were capable of converting a large part of the dosed 800 ppm_{C1} OME to CO₂ already between 80 and 100 °C, thus releasing the corresponding energy. However, the temperatures, which are required to convert more realistic OME concentrations of a few thousand ppm, could not be investigated within the scope of this first short study. It should be noted that none of the two DOCs tested were able to convert all of the OME to CO₂. Both samples showed traces of OME and thus formaldehyde downstream of the catalysts, despite their very high oxidation activity. An optimized DOC comprising an OME hydrolysis catalyst (based on zeolites due to their high hydrolysis activity already at low temperatures) and a PGM-based oxidation catalyst appears to be most promising solution. Another option is the combination of an upstream VWT catalyst for OME hydrolysis with a downstream DOC for subsequent oxidation and temperature increase.

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