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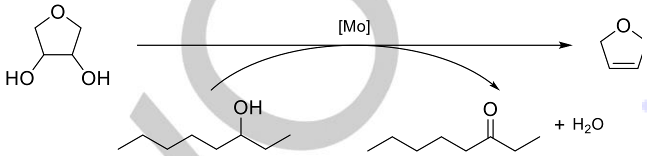
Supported molybdenum catalysts for the deoxydehydration of 1,4-anhydroerythritol into 2,5-dihydrofuran

Lennart Sandbrink,[a][d] Klaus Beckerle,[b][d] Isabell Meiners,[a] Rebecca Liffmann,[b] Khosrow Rahimi,[c] Jun Okuda[b]* and Regina Palkovits[a]*

Abstract: Efficient deoxygenation strategies are crucial for the valorization of renewable feedstocks. Deoxydehydration (DODH) enables the direct transformation of two adjacent hydroxyl groups into a double bond. Supported molybdenum-based catalysts were utilized for the first time in DODH. MoO_x/TiO_2 possesses superior catalytic activity compared to common molybdenum salts. The catalyst efficiently converted 1,4-anhydroerythritol into 2,5-dihydrofuran in the presence of 3-octanol as reducing agent showing high reproducibility and stability.

Introduction

In contrast to petrochemical value chains, deoxygenation reactions are the major pathway to valorize renewable feedstock molecules into bio-based products.[1] The deoxydehydration (DODH) has evolved to be an efficient alternative to established deoxygenation routes. It offers direct access to olefins from diols, which are abundant functional groups in biomass-related feedstocks.[2] The first catalytic deoxydehydration was described by Cook and Andrews in 1996.[2] Starting from 2008 several researchers expanded the reaction class to the high potential that it offers today.[3] The reaction can be carried out with molecular rhenium-,[3] molybdenum-[4] or vanadium-based[5] catalysts, which in combination with a stoichiometric reductant enable the transformation of various diols into olefins. During the past years, the reductant, catalyst and substrate scope has been expanded drastically.[6] To develop DODH further, supported rhenium catalysts were presented to obtain heterogeneous systems. [7] With ReO_x/TiO_2 we recently reported a stable heterogeneous DODH-catalyst.[7b] Further heterogeneous systems were published by Nakagawa and Tomishige. They applied ceria supported Rhenium catalysts promoted with Au or Pd for the selective deoxydehydration or hydrodeoxygenation of various diols and polyols in the presence of hydrogen gas as reducing agent.[8] However, an economic process with such rhenium catalysts remains certainly challanging. In order to facilitate a broader application of the reaction class, we report here the first supported molybdenum catalysts for DODH. In the following studies the DODH of 1,4-anhydroerythritol (1,4-AE) to 2,5-dihydrofuran (2,5-DHF) was used as model reaction (Scheme 1). 1,4-AE can be easily derived from erythritol,[9] which is a fermentation product of sugars.[10] Previous studies already showed that 1,4-AE is a promising bio-based intermediate for the production of C_4 chemicals.[6d,,8a,11]



Scheme 1. Scheme of the model reaction: DODH of 1,4-AE into 2,5-DHF.

Results and Discussion

In the current study, commercially available molybdenum compounds including $Mo(CO)_6$ [**1**], $MoCl_3$ [**2**], MoO_2Cl_2 [**3**], $Na_2MoO_4 \cdot 2H_2O$ [**4**], Na_2MoO_4 [**5**], $(NH_4)_2MoO_4$ [**6**], MoO_3 [**7**], $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ [**8**], and were initially examined in the DODH of 1,4-anhydroerythritol to 2,5-dihydrofuran using 3-octanol as reducing agent and solvent (Table 1). During the reaction, 3-octanol is oxidized to 3-octanone. Under standard reaction conditions (5 mol.% Mo, 10 equiv. 3-octanol relative to substrate 200 °C, 18 h), most of the compounds were found to be active in the test reaction. Although the highest yields of 2,5-dihydrofuran were observed for compounds with an oxidation state (OS) of +VI for molybdenum, the oxidation states 0 and +III were found to be active as well. $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ [**8**] facilitated the highest overall conversion and yield of the DODH-product. The reaction mechanism of the Molybdenum-catalyzed DODH was proposed to be based on the condensation of MoO_3 with the diol Afterwards coordination and transfer hydrogenation of the reducing agent and the final extrusion of the DODH-product take place.[12] Next to the DODH-product 2,5-DHF, high amounts of the ketal, formed by condensation of 3-octanone and 1,4-anhydroerythritol, were obtained as the major side product. The dehydration of 3-octanol to 2- and 3-octene occurred as side reaction (see supporting information (SI), Scheme S1 and S2). The gap in the mass balance is assumed to be based on condensation reactions producing high boiling products. These products were not detectable with the applied GC-MS-analysis due to lack of volatility (compare NMR-analysis in SI, Figure S8 and S9).

As already described by the group of Fristrup in 2014 for the DODH of 1,2-tetradecanediol in dodecane using 3-octanol as reductant, the Mo(+IV) compound sodium molybdate [**4**] was inactive in DODH.[4] The use of anhydrous sodium molybdate [**5**] showed no increase in catalyst activity. Although the authors explained the inactivity of Na_2MoO_4 by the lack of solubility in the dodecane solvent,[4] we assume that also the type of active molybdate species can play a key role as discussed for Table 2.

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Table 1. Product distribution of homogeneous reference catalysts in the test reaction 1,4 anhydroerythritol (1,4-AE) to 2,5 dihydrofuran (2,5-DHF).[a]

Catalyst (OS)	X (1,4-AE) [%]	Y (2,5-DHF) [%]	Y (ketal) [%]	Y (octenes)[b] [%]
-	9	0	2	0
$Mo(CO)_6$ [1] (0)	36	21	6	6
$MoCl_3$ [2] (+III)	75	22	15	16
MoO_2Cl_2 [3] (+IV)	60	28	10	11
$Na_2MoO_4·2H_2O$ [4] (+IV)	9	0	2	0
Na_2MoO_4 [5] (+IV)	11	1	1	0
$(NH_4)_2MoO_4$ [6] (+IV)	55	27	10	3
MoO_3 [7] (+VI)	91	29	20	5
$(NH_4)_6Mo_7O_{24}·4H_20$ [8] (+VI)	83	38	19	3

[a] Conditions: 0.4 mmol 1,4-anhydroerythritol, 5 mol% Mo and 10 eq. 3-octanol relative to substrate, 200 °C, 18 h. Conversion and yields determined by NMR spectroscopy. [b] Based on 3-octanol.

Table 2. Product distribution of supported Mo-catalysts and mixtures in the test reaction 1,4 anhydroerythritol (1,4-AE) to 2,5 dihydrofuran (2,5-DHF).[a]

Catalyst (Mo content [%])[b]	X (1,4-AE) [%]	Y (2,5-DHF) [%]	Y (ketals) [%]	Y (octenes)[c] [%]
$Na_2MoO_4·2H_2O/TiO_2$ [**4a**] (4.8)	27	1	0	<1
MoO_x/TiO_2 ($Na_2MoO_4·2H_2O$)[d] [**4b**] (5.1)	39	5	1	<1
$(NH_4)_6Mo_7O_{24}·4H_20/TiO_2$ [**8a**] (4.9)	100	48	3	15
MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}·4H_20$)[e] [**8b**] (4.7)	94	55	9	48
In-situ mixture [**4c**]: $Na_2MoO_4+TiO_2$	19	4	0	3
In-situ mixture [**8c**]: $(NH_4)_6Mo_7O_{24}·4H_20+TiO_2$	100	76	10	16
TiO_2 [**9**]	14	2	1	1

[a] Conditions: 0.4 mmol 1,4-anhydroerythritol, 5 mol% Mo and 10 equiv. 3-octanol relative to substrate, 200 °C, 18 h. Conversion and yields determined by NMR spectroscopy. [b] Determined by ICP-OES. [c] Based on 3-octanol. [d] Mo-precursor: $Na_2MoO_4·2H_2O$, reduction conditions: 3 h, 300 °C, 2 °C/min. [e] Mo-precursor: $(NH_4)_6Mo_7O_{24}·4H_20$, reduction conditions: 3 h, 300 °C, 2 °C/min.

In the presence of $MoCl_3$ [**2**] and MoO_2Cl_2 [**3**], the yield of octenes increased due to hydrochloric acid formation during the reaction catalyzing the dehydration of 3-octanol.

Ammonium heptamolybdate [**8**] and sodium molybdate [**4**] were selected as precursors for the impregnation on a support material. Titanium dioxide enabled already high activity of supported rhenium catalysts in DODH[7b] and was selected as support. A fraction of the impregnated catalysts was directly tested in the DODH of 1,4-anhydroerythritol. The other fraction was reduced to lower oxidation state of molybdenum oxides (MoO_x) in H_2-atmosphere at 300 °C prior to reaction. Generally, the use of ammonium heptamolybdate [**8a-c**] increased the conversion and yield of the DODH-product compared to the other commercially available molybdenum salts (Table 2). Catalysts based on sodium molybdate [**4a-c**] possessed low catalytic activity. Raman spectra of the two reduced catalysts [**4b**, **8b**] showed a characteristic band between 890 and 960 cm^{-1}, which is attributed to the stretching modes of the terminal Mo=O bond (Figure 1). The wavenumber of the band increases with the degree of polymerization. Therefore, the lower wavenumber for the sodium molybdate based catalysts [**4a**,**b**] is explained by the formation of isolated MoO_4^{2-} species on the surface. The use of ammonium heptamolybdate as a precursor [**8a**,**b**] results in polymerized clusters on the catalyst surface,[13] associated with a higher activity for DODH in comparison to isolated molybdate species.

The influence of reduction on the product yield was rather low, but octene formation increased, indicating more pronounced acidic properties of the reduced catalysts. Raman spectra of the reduced materials showed an increase of the wavenumber of the bands.

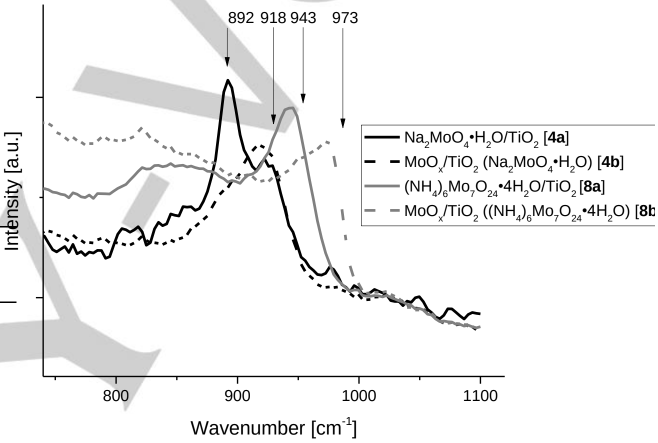

Figure 1. Raman spectra of impregnated [**4a**, **8a**] and reduced [**4b**, **8b**] molybdate catalysts in the region of characteristic Mo-oxo bonds (anatase background subtracted).

Pure titanium dioxide [**9**] is inactive in DODH. However, an *in-situ* mixture of titanium dioxide and ammonium heptamolybdate [**8c**] resulted in higher yields of 2,5-dihydrofuran than the supported catalysts formed prior to reaction [**8a**,**b**]. Similar to the unreduced catalyst [**8a**], the mixture [**8c**] resulted in lower octene formation compared to the reduced one [**8b**].

The most active catalysts MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}·4H_2O$) [**8b**] and *in-situ* mixture [**8c**] $(NH_4)_6Mo_7O_{24}·4H_2O+TiO_2$ were further examined in terms of reproducibility and recyclability (Figure 3 and 4). For each of the systems three independent test reactions were performed, showing good reproducibility for both of them (see SI, Figure S1 and S2).

After the reaction, two of the catalysts were recycled and subjected to four consecutive runs. The impregnated and reduced catalyst [**8b**] exhibited good reproducibility in the DODH reaction. Recycling of the *in-situ* formed [**8c**] $(NH_4)_6Mo_7O_{24}·4H_2O+TiO_2$ features higher deviations in conversion and yield of 2,5-dihydrofuran. However, excellent recyclability emphasizes an efficient *in-situ* formation of a supported molybdenum catalyst. The most noticeable difference of the both recycling series could be observed in the formation of octenes. While octene formation remained constantly low for the recycling of the *in-situ* mixture [**8c**], the use of the impregnated and reduced catalyst [**8b**] resulted in an initially high yield and a

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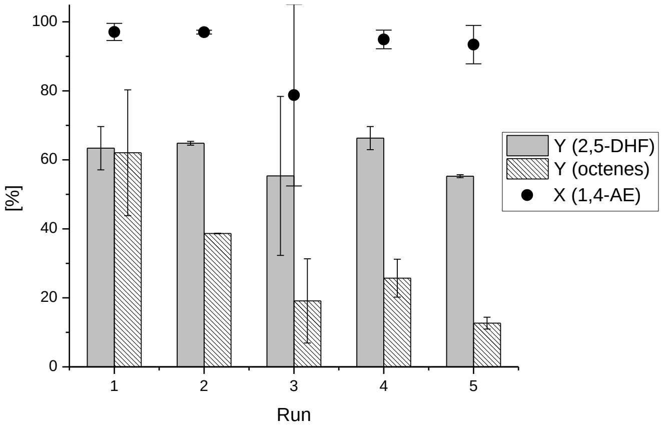

Figure 3. Recycling of MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**]. Conditions: 0.4 mmol 1,4-anhydroerythritol, 5 mol% Mo in the starting catalyst and 10 equiv. 3-octanol relative to substrate, 200 °C, 18 h. Conversion and yields determined by NMR spectroscopy. Octene-yield based on 3-octanol.

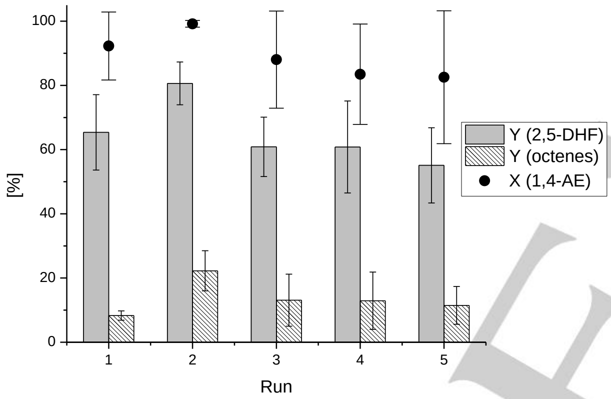

Figure 4. Recycling of the *in-situ* formed mixture [**8c**] $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$+$TiO_2$. Conditions: 0.4 mmol 1,4-anhydroerythritol, 5 mol% Mo in the starting catalyst, and 10 equiv. 3-octanol relative to substrate, 200 °C, 18 h. Conversion and yields determined by NMR spectroscopy. Octene-yield based on 3-octanol.

subsequent decrease of the octene formation to a level comparable to the in-situ formed catalyst system [**8c**]. These results suggest a higher acidity for the impregnated catalyst [**8b**] than for the in-situ formed mixture [**8c**], dropping for every run in the model reaction. Further studies will focus on the role of acidity for supported Mo catalysts in DODH. Overall, the recycling experiments indicate a high stability of the catalyst material, which could be confirmed by ICP-MS measurement. The leaching of Mo and Ti during every run was below 0.5 % (see SI, Table S2 and S3). A hot filtration test supports that the reaction does not proceed in homogeneous phase as no further 2,5-DHF is generated after filtration (see SI, Figure S10). Furthermore, freshly prepared MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**] and both catalysts after 5 runs were analyzed with SEM-EDX confirming a comparable fine dispersion of Mo species on the surface of the support (Figure 5). A closer look emphasizes a slight decrease of the Mo/Ti ratio for freshly prepared MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**] and after 5 runs further supporting a high stability upon recycling (SI, Figure S4-S7). EDX mapping of catalysts after recycling reveals a slightly lower Mo/Ti ratio and a more uniform Mo distribution in case of *in-situ* mixture [**8c**] $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$+$TiO_2$ (Figure 5 and SI, S6 and S7, Table S1). Consequently, the nature of Mo aggregation as well as its uniform and high dispersion are potentially responsible for the superior catalytic activity of this material system.

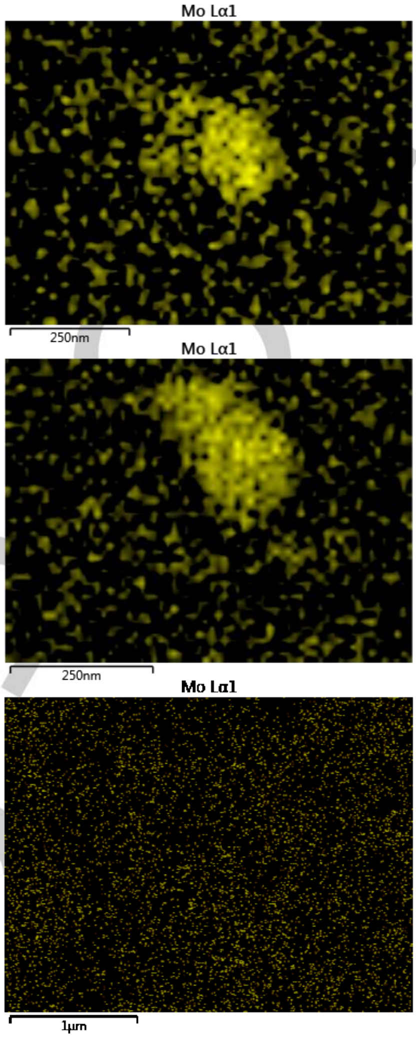

Figure 5. SEM-EDX mapping: Distribution of Mo on the surface of freshly prepared MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**] (above), as well as MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**] (middle) and *in-situ* mixture [**8c**] $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$+$TiO_2$ (below) after 5 runs (the corresponding spectra are shown in SI).

Conclusions

In conclusion, we have developed a stable and efficient supported molybdenum catalyst for the DODH of 1,4-anhydroerythritol to 2,5-dihydrofuran. The catalytic activity of the best currently known molybdenum based DODH-catalyst,

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ammonium heptamolybdate, was increased noticeably by using titanium dioxide as support material. The *in-situ* mixture of ammonium heptamolybdate with titanium dioxide [**8c**] resulted in good catalytic activity, but moderate reproducibility. A higher reproducibility was observed with MoO_x/TiO_2 ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) [**8b**], which could be recycled five times without significant decrease of activity. Additionally, Raman-spectroscopic and SEM-EDX investigations provided a first insight in the nature of active species with superior performance of polynuclear molybdenum compounds compared to mononuclear ones. Additionally, uniform Mo dispersion appears to be key.

Experimental Section

All experiments were performed under dry argon atmosphere using Schlenk or glovebox techniques. NMR spectra were recorded on a Bruker Avance II 400 MHz at 23 °C (^1H: 400 MHz).

Anhydroerythritol, 3-octanol and mesitylene were purchased from Sigma Aldrich and used after drying over molecular sieve (4 Å) and degassing. Molybdenum compounds were purchased from Sigma Aldrich and used as received. TiO_2 ST61120 was purchased from Saint-Gobain NorPro and pestled before use.

General procedure for catalyst preparation

The appropriate amount Mo-precursor was dissolved in water (20 mL per gram support). The appropriate amount of the support was added slowly and the mixture was stirred 24 h at rt. Afterwards the solvent was removed by evaporation and the product was dried at 120 °C for 24 h. Half of the product was reduced under H_2-flow for 3 h at 300 °C.

General procedure for deoxydehydration reactions

A thick-walled glass tube with a screw cap was charged in the glovebox sequentially with the catalyst (18 µmol of Mo), anhydroerythritol and 3-octanol in the given ratio. A magnetic stirring bar was added and the mixture was stirred for 10 minutes at ambient temperature before it was inserted into a preheated oil bath. After the given reaction time, the glass tube was taken from the heating bath and cooled down with cold water. Mesitylene (23 µL) was added as an internal standard and the mixture was vigorously shaken. Upon homogenization, an aliquot (roughly 0.05 mL) of the mixture was taken and mixed with 0.5 mL $CDCl_3$. The mixture was analyzed by ^1H-NMR spectroscopy.

Recycling tests

A DODH reaction was performed as described in the general procedure. For the first run, two glass tubes were charged with catalyst (38 mg) or a mixture of ammonium heptamolybdate (3.2 mg) and titanium dioxide (38 mg) under air and brought into a glovebox after exchanging atmosphere to argon. A stock solution of anhydroerythritol and 3-octanol (560 mg: 20 equiv. of anhydroerythritol per molybdenum, anhydroerythritol:3-octanol 1:10) and a magnetic stirring bar were added and the cap was closed tightly. The tubes were placed into a preheated oil bath at 200 °C and kept stirring for 18 h. Afterwards, the tubes were taken out of the oil bath cooled down into an ice/water bath. 1 mL $CDCl_3$ was added for dilution and 23 µL mesitylene was added as internal standard. After filtration aliquots of the resulting solution were taken and analyzed by ^1H NMR spectroscopy. The retained solids were washed with chloroform (2 times 2 mL), acetone (2 mL) and again chloroform (2 mL). The solids were dried under air for 1 h at ambient temperature and at least 3 h at 130 °C. The residues were weighed and the reaction scales were recalculated accordingly before re-subjecting them to the procedure.

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Keywords: molybdenum • heterogeneous • deoxydehydration • biomass • diol

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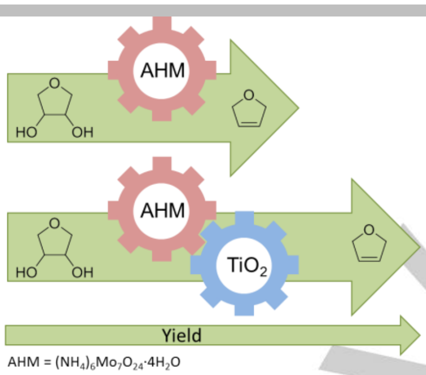
More than the sum: We present the first supported molybdenum catalyst for deoxydehydration. Interestingly, the titania supported Mo catalyst outperforms the molecular counterparts.



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