Advanced Membrane Reactors in Energy Systems
A Carbon-Free Conversion of Fossil Fuels

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Abstract

The purpose of this project is to develop hydrogen and CO_2 selective membranes to allow combination of natural gas reforming with H_2 or CO_2 separation in separation enhanced reactors, i.e. membrane reactors, for carbon-free hydrogen production or electricity generation. To achieve this, the project comprises three distinct tasks: system and reactor analysis, membrane materials research and catalysis.

The results of the system and reactor analysis demonstrate that doubling the target permeation (derived from H_2-selective WGS-MR, Water-Gas-Shift-Membrane Reactor) of CO_2 selective WGS-MR already leads to a competitive advanced membrane reactor configuration. Moreover, the high CO_2 purity as well as the flexibility with respect to syngas conversion and CO_2 separation in combination with the reduced hydrogen loss due to combustion with traces of oxygen, are important advantages for implementation of the CO_2-selective WGS-MR.

Materials science has led to the following findings. Structure, composition and decomposition pathway of Mg-Al Hydrotalcite have been determined. The Mg/Al ratio is found to be 1.8, the net formula is $Mg_{0.64}Al_{0.36}(OH)_2(CO_3)_{0.18} \cdot 1.0\ H_2O$. At about $250^{o}C$ the carbonate-water layer disappears and with this the presupposed transport path for CO_2 through a dense hydrotalcite membrane. Porous hydrotalcite based membranes where the transport and selectivity are based on affinity of CO_2 to the micro pore walls are now the focus of R&D at ECN. Coatings based on nano particulate dispersions or sol gel synthesized particles and sensitizing microporous supports are the three routes to achieve our goal.

The catalysis work revealed that specific nobel metal and FeCr based WGS catalysts prove to be stable for at least 200 h, under experimental conditions without sulphur compounds.

Introduction

A sustainable use of fossil fuels in the future will undoubtedly make use of concepts, where the energy content of the fossil fuel is first transferred to hydrogen, followed by the conversion to the desired energy form. The driving force for these concepts is the possibility of capturing CO_2 elegantly, while using the favorable thermodynamics to increase the efficiencies of fossil fuel conversion. We, ECN and TU-Delft, have

identified membrane reactors as a game changing technology for highly efficient conversion of fossil fuels to carbon free energy carriers.

The purpose of this project is to develop hydrogen and CO_2 membranes to allow combination of natural gas reforming with H_2 or CO_2 separation in separation enhanced reactors, i.e. membrane reactors, for carbon-free hydrogen production or electricity generation. These devices offer multiple advantages, such as eliminating the requirement of water gas shift reactors with associated costs reductions; offering higher conversion efficiencies at lower temperatures; and decreasing primary energy use for CO_2 separation/capture associated with electricity generation.

Background
The steam reforming and the water gas shift equilibriums are key reactions for the production of hydrogen from fossil fuels:

$$CH_4 + H_2O \leftrightarrows CO + 3H_2 \tag{1}$$
$$CO + H_2O \leftrightarrows CO_2 + H_2 \tag{2}$$

By removing either CO_2 or hydrogen from the reaction mixture, the equilibrium can be shifted to the product side. Effectively, this can lower the reaction temperature and improve the purity of the product. In conventional, hydrogen production from natural gas, the steam-reforming step is followed by two water gas shift (WGS) reactors. When separating either CO_2 or hydrogen inside the reforming reactor, both the WGS steps can be eliminated. This implies that separation-enhanced techniques can also lead to investment costs reductions. Hydrogen or CO_2 separation is a flexible technique that can be used in hydrogen production from natural gas, but also can replace the WGS section of an IGCC or Biomass gasification plant. These techniques are especially suited for CO_2 capture, because the production of pure hydrogen and CO_2 streams is intrinsic to separation-enhanced reactors.

The combination of separation and reaction, as foreseen in membrane rectors, offers higher conversion of the reforming reactions at lower temperatures due to the removal of hydrogen or CO_2 from these equilibrium reactions, as shown in equations 1 and 2. For instance, in case natural gas reforming for carbon free hydrogen production, the use of membrane reactors will result in significantly lower operation temperatures (400 - 500°C) and higher efficiencies 85 - 90 instead of 75%.[1] In fact membrane reactors allow for low-irreversibility production and conversion of hydrogen to another energy form with integrated CO_2 capture.

Membrane reformers/reactors can be integrated in power generation systems but also in central heating devices. Our assessment studies clearly showed that in a more integrated approach of electricity production and CO_2 capture, using high-temperature membrane reactors will result in a substantially lower primary energy use for the CO_2 separation/capture [2]. Besides that, the low operation temperature of the membrane reactor creates possibilities for so-called chemical recuperation, compensating part of the CO_2 capture efficiency penalty.

The tasks defined within this project are:

Task 1   System analysis and thermodynamic evaluations   Executed by ECN
Task 2   Hydrogen membrane research and development   Executed by TUD
Task 3-a Hydrotalcite CO_2 membranes research and development Executed by ECN
Task 3-b Ionic liquids CO_2 membranes research and development Executed by TUD
Task 4   Catalyst screening   Executed by ECN
Task 5   Reactor modeling and design   Executed by ECN

   Tasks 1, 4 and 5 pertain to both the hydrogen and carbon dioxide membrane cases.

Results

System and reactor analysis:

   Two advanced membrane reactor configurations were assessed wit respect to implementation in an Integrated Gasification Combined Cycle (IGCC) with pre-combustion CO_2 capture. The advanced membrane reactors comprise a H_2- and CO_2-selective Water-Gas-Shift Membrane Reactor (WGS-MR), in which the separation enhances the equilibrium limited water-gas-shift reaction. Dry-fed coal gasification was selected for these detailed system assessments. The assessments were performed with AspenPlus combined with the in-house developed membrane model [3], as well as 'Exercom' that facilitates second law analysis.

   Sensitivity analysis identified the most important variables that govern the specific electric loss, when a H_2-selective WGS-MR is implemented in an IGCC. These are the steam/CO-ratio at the inlet of the pre-WGS reactor and the pressure of the N_2 sweep stream. Figure 1 displays that the steam consumption should be minimised since steam is applied to generate electricity in the bottoming cycle, whereas the membrane sweep pressure should be equal to the inlet pressure of the gas turbine combustion chamber, being 23 bars.
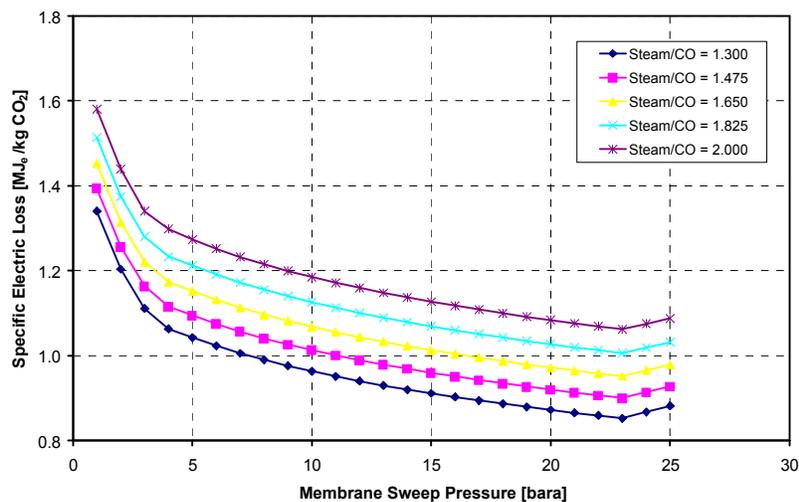

Figure 1 Specific electric losses for an IGCC with H_2-selective WGS-MR at variable membrane sweep pressures and Steam/CO-ratios

A similar sensitivity analysis was performed for implementation of a CO_2-selective WGS-MR also implemented in an IGCC. The variables that govern the specific electric loss in this case are the steam/CO-ratio at the inlet of the pre-WGS reactor and the membrane sweep flow. The membrane is swept with low pressure steam (6 bara); sweeping with intermediate pressure steam (25 bara) and expanded steam (0.03 bar) were also evaluated but proved to be less efficient (see). Figure 2 displays that both the steam consumption for the pre-WGS and the sweep flow should be minimised. The results of the system assessments are presented in (Table I). The overall efficiency for the reference case (without CO_2 capture) is relatively high in comparison with reported values, which is to ascribe to the supposed application of H-class gas turbine combined cycles. The results indicate that the H_2-selective WGS-MR case results in the lowest efficiency penalty (P_{feed} = 25 bara), whereas the CO_2-selective WGS-MR case results in the highest efficiency penalty (P_{feed} = 25 bara; P_{sweep} = 6 bara). The reference case with CO_2 capture (Selexol) case results in an absolute efficiency penalty of 7.7%, which corresponds with values reported in literature [4].
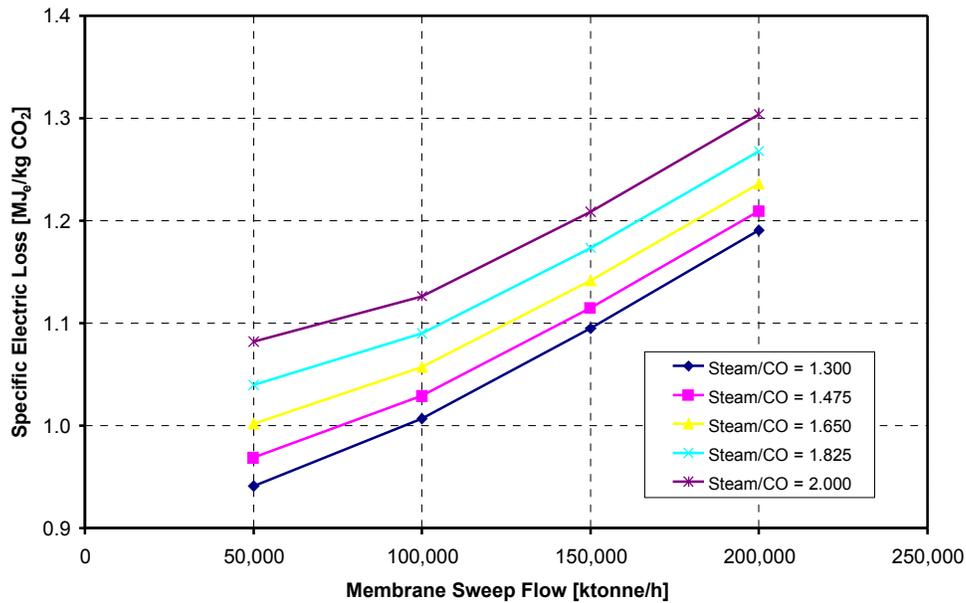

Figure 2 Specific electric loss for an IGCC with CO_2-selective WGS-MR at variable membrane sweep flows (steam at 6 bara) and steam/CO-ratios

Table I Results system assessments for various cases and membrane surface area of 10,000 m^2

P_{feed} [bara]	P_{sweep} [bara]	F_{sweep} [kg/s]	Permeance[7] [$\times 10^{-6}$ mol m^{-2} s^{-1} Pa^{-1}]	Φ_{mem} [$\times 10^{-5}$ mol m^{-2} s^{-1}]	$P_{electric}$ [MW$_e$]	$\eta_{energetic}$ [-]	CCR [-]	Specific Loss [MJ$_e$/kg CO$_2$]
Without CO$_2$ capture								
-	-	-	-	-	500.0	47.5	-	-
CO$_2$ Capture with high & low temperature WGS and Selexol								
-	-	-	-	-	419.2	39.8	91.7	0.885
CO$_2$ Capture with high temperature pre-WGS, H$_2$-selective WGS-MR including cryogenic distillation								
25	23	94.4	2.0	12.8	427.5	40.6	83.1	0.853
40	23	94.4	2.0	18.5	428.6	40.7	88.7	0.791
CO$_2$ Capture with high temperature pre-WGS and CO$_2$-selective WGS-MR								
25	0.03	13.9	2.0	12.9	379.5	36.0	85.5	1.416
25	6	55.6	2.0	11.7	411.3	39.1	74.9	1.191
25	25	55.6	2.0	6.8	456.4	43.3	34.4	1.276
25	6	27.8	4.0	11.6	435.2	41.3	76.4	0.852
40	6	13.9	2.0	12.6	430.1	40.9	83.2	0.844
40	6	13.9	4.0	14.0	424.9	40.4	92.4	0.816

Application of CO$_2$-selective WGS-MR appears to be less efficient than the H$_2$-selective WGS-MR case. However, it should be noted that the CO$_2$ product stream obtained from the retentate of the H$_2$-selective WGS-MR contains a number of impurities. These impurities entail: N$_2$ (applied to pressurize and transport pulverized coal to the gasifier, and coal-bound nitrogen), CO (unconverted), H$_2$ (not permeated) and CH$_4$ (gasification product, small quantity). The presence of these impurities makes an additional separation step such as cryogenic distillation inevitable (cost).

Moreover, a small amount of oxygen (typically 1.5 mol%) is present in N$_2$ obtained from the ASU, which is applied as sweep flow within the H$_2$-selective WGS-MR case. Combustion of hydrogen with this oxygen on the permeate side of the membrane was neglected during the system assessments; however this could result in unacceptable high temperature gradients across the counter-current membrane configuration. Subsequently, this results in a reduced H$_2$ availability for the combustion chamber of the gas turbine.

The application of low pressure steam to sweep the CO$_2$-selective WGS-MR results in the lowest specific electric loss compared with application of intermediate pressure or expanded steam. Although it appears that implementation of H$_2$-selective WGS-MR is still more efficient than CO$_2$-selective WGS-MR.

Subsequent sensitivity analyses demonstrated that doubling the target permeance leads to comparable specific losses for both WGS-MR cases, the CO$_2$-selective WGS-MR having a slight higher efficiency and a slightly lower carbon capture ratio. Increasing the feed pressure to 40 bara more significantly reduces the specific electric loss for the CO$_2$-selective WGS-MR.

It can be readily concluded from the results that something like a negative correlation exists between efficiency penalty and carbon capture ratio (CCR). Optimization of the

CCR generally results in a lower efficiency. Membrane properties optimization, i.e. increasing the permeance, is the only way to break out of this *status quo*.

The results of the system assessments demonstrate that doubling the target permeation (derived from H_2-selective WGS-MR) of CO_2 selective WGS-MR already leads to a competitive advanced membrane reactor configuration. Moreover, the high CO_2 purity as well as the flexibility with respect to syngas conversion and CO_2 separation in combination with the reduced hydrogen loss due to combustion with traces of oxygen, are important advantages for implementation of the CO_2-selective WGS-MR

Membrane materials research

The research on hydrotalcite, $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$, as a possible CO_2 selective membrane material, has been pursued. The stability window of hydrotalcites, as to temperature, CO_2 and H_2O partial pressures as well as the compositional differences between various commercial and in-house synthesized materials have been determined. To this end, *in-situ* XRPD (X-Ray Powder Diffraction) and DRIFT (Diffuse Reflectance Infrared Fourier Transform) as well as NPD (neutron powder diffraction), TGA-MS (Thermogravimetry-Mass Spectroscopy) and SEM-EDX (Scanning Electron Microscopy-Energy Dispersive X-ray analysis) measurements have been performed.

A comparison between the commercial materials and the in-house produced ones has been performed with XRD. From this it can be deduced that the commercial materials are poorly crystalline compared to the in-house produced ones. Furthermore, the more the Mg/Al ratio is closer to 2 the more crystalline the materials are. Since the number of electrons of Mg^{2+} and Al^{3+} are equal, XRPD can not be used to find the ratio between these ions, however, with NPD one can. Three samples of different Mg/Al ratios (90%, 50% and 25%) have been analyzed at the GEM beam line at ISIS, UK.
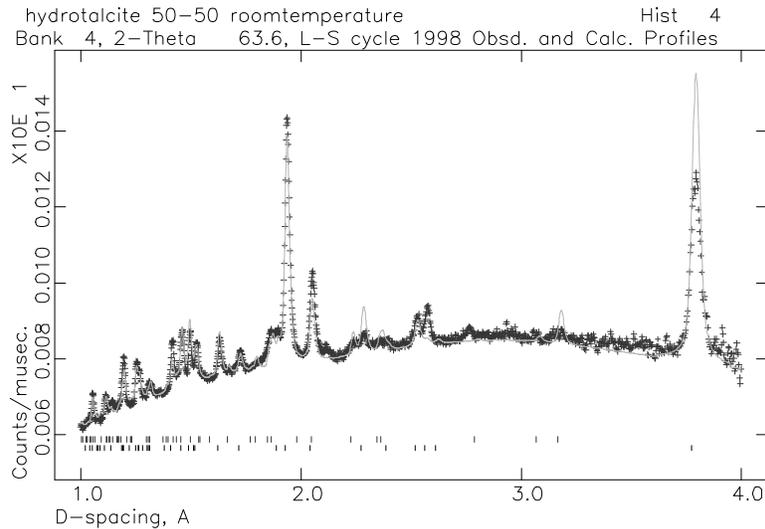

Figure 3 Neutron powder diffraction pattern with fit of Mg50 (starting materials: Mg/Al=1). The upper tick marks pertain to Boehmite the lower ones to Hydrotalcite.

The diffraction pattern (Figure 3) could be refined using the GSAS program suite, in which the carbonate ion and water molecule are treated as rigid bodies. The Mg/Al ratio is assessed to be about 1.8, yielding the net formula $Mg_{0.64}Al_{0.36}(OH)_2(CO_3)_{0.18} \cdot 1.0\ H_2O$. Deviations from this value yield either Al rich (Boehmite) or Mg rich (Hydromagnesite) impurity phases. This confirms our previous presumption that there is only a small compositional window for hydrotalcite with respect to the cations.

The water-gas-shift reaction takes place at about 400°C. TGA-MS has been performed on the commercial material MG61 to identify the decomposition steps. Four decomposition steps could be observed. The first step at ≈100°C corresponds to adsorbed water, the second one at ≈240°C represents the interlayer water disappearing, the third one at ≈310°C represents the hydroxides decomposing into oxides and the fourth one at ≈450°C represents the last hydroxides and carbonates decomposing into oxides. To characterize the structural evolution upon decomposition, in-situ XRPD has been performed on the same material (Figure 4).
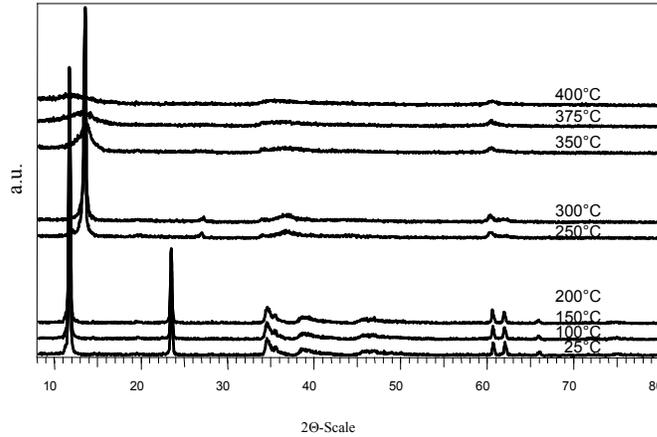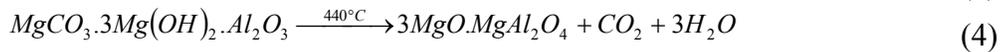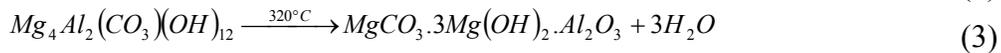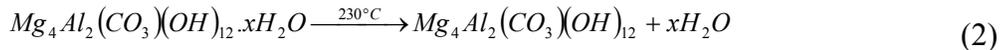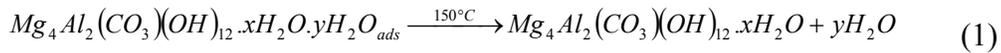

Figure 4 In-situ XRPD pattern of MG61 as a function of temperature.

Figure 4 confirms the three first steps found with TGA-MS. Between 200°C and 250°C, there is a shift towards smaller d-spacing, indicative of the collapse of the interlayer at ≈240°C. Between 300°C and 350°C the crystallinity of the material is strongly decreasing, which confirms that the hydroxides are decomposing into oxides. More detailed information on the process has been obtained using DRIFT. This technique permits to perform infrared spectroscopy under different gas atmospheres and temperature. Combination of XRPD, TGA-MS and DRIFT yields the following decomposition pathway:

$$Mg_4Al_2(CO_3)(OH)_{12} \cdot xH_2O \cdot yH_2O_{ads} \xrightarrow{150°C} Mg_4Al_2(CO_3)(OH)_{12} \cdot xH_2O + yH_2O \qquad (1)$$

$$Mg_4Al_2(CO_3)(OH)_{12} \cdot xH_2O \xrightarrow{230°C} Mg_4Al_2(CO_3)(OH)_{12} + xH_2O \qquad (2)$$

$$Mg_4Al_2(CO_3)(OH)_{12} \xrightarrow{320°C} MgCO_3 \cdot 3Mg(OH)_2 \cdot Al_2O_3 + 3H_2O \qquad (3)$$

$$MgCO_3 \cdot 3Mg(OH)_2 \cdot Al_2O_3 \xrightarrow{440°C} 3MgO \cdot MgAl_2O_4 + CO_2 + 3H_2O \qquad (4)$$

Since the transport path for CO_2 through the membrane, i.e. the carbonate-water layer, does not exist anymore, hydrotalcites can not be used as dense membranes. This leaves only the porous option for which the separation is based on affinity of CO_2 to the pore walls, thereby blocking the H_2 molecules (Figure 5).
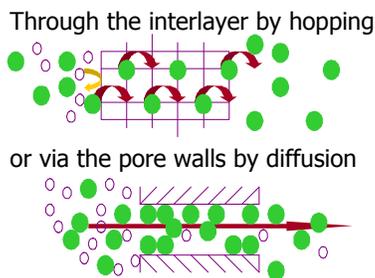

Figure 5 Transport mechanisms for CO_2 through a dense (upper) and porous (lower) membrane

Preparation options for enhanced affinity membranes based on hydrotalcitic material comprise thin layer deposition from nano sized particles in dispersion or via sol gel synthesis on a porous support. Chemical modification of micro porous alumina with magnesium salts is a third option. Pioneering experiments have been performed on the synthesis of nano particulate dispersions (Figure 6). Dynamic light scattering showed that apart from these 10-15 nm particles large agglomerates are present that appear really hard to break down.
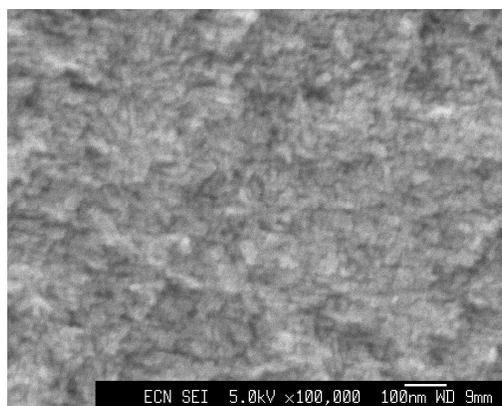

Figure 6 SEM picture of agglomerated 10-15 nm sized hydrotalcite particles

Catalyst screening

The work planned for the first part of the catalysis task, is the screening of catalysts to assess the applicability of commercially available catalysts in either CO_2- or hydrogen-separating membrane reactors. In the previous reporting period the results on the four commercial Methane Steam Reforming (MSR) catalysts were reported. Results obtained so far on WGS catalysts are presented in the following.

Experimental program:

Based on the expected process conditions in an actual membrane shift reactor, the following reaction conditions have been selected:

- Gas phase composition: 10% CO, 19% H_2O, 20% CO_2, 20% H_2, 31% N_2.
- Average operating temperature: 450°C.
- Expected operating pressure: 25 barg.
- Sulphur present to a maximum of 20 ppmv of H_2S.

Based on the experimental setups available, the screening programme has been divided into four subsets:
1. Activity measurement at atmospheric pressure, 300–500°C
2. Activity measurement at maximum available pressure, 300–500°C
3. Stability measurement at atmospheric pressure, 450°C
4. Stability measurement at atmospheric pressure in presence of 20 ppmv H_2S, 450°C

Given these operating conditions, two types of catalysts will be considered: high-temperature WGS catalysts based on Fe_2O_3/Cr_2O_3 and WGS catalysts based on noble metals. Low temperature WGS catalysts are discarded in view of the relatively high operating temperature. Six commercially available catalysts have been selected for testing. From previous experiments it is known that commercial Fe_2O_3/Cr_2O_3 catalysts show identical performance and it is unlikely that these materials are affected by membrane specific operating conditions or H_2S. However, most of these catalysts contain CuO as well, which could present a problem in presence of H_2S. Therefore two Fe_2O_3/Cr_2O_3 catalysts have been selected: one with CuO and one without. Four noble metal catalysts are tested. Two have been selected for their reported activity. The other two have been selected for their claimed sulphur tolerance.

1. ICI 71-5M, commercial HTS catalyst ("FeCrCu")   ($Fe_2O_3/Cr_2O_3/CuO$)
2. Haldor Topsøe SK 201-2, commercial HTS catalyst ("FeCr")   (Fe_2O_3/Cr_2O_3)
3. Umicore W-Type noble metal ("NM1")
4. Süd-Chemie FCS12 noble metal ("NM2")
5. NexTech ZDC-Pt sulphur-tolerant commercial noble metal ("NM3")
6. NexTech ZDC-Rh sulphur-tolerant commercial noble metal ("NM4")

Activity measurements at atmospheric pressure

To compare the activity of catalysts, activity measurements have been done in a temperature range of 300–500°C. The noble metal catalysts have been diluted as powders with 95% of high purity α-Al_2O_3, to a total of 100 mg of sieve fraction. The Fe_2O_3/Cr_2O_3-based catalysts have been tested as 200 mg of undiluted sieve fraction. Both types of sample were tested with a total flow of 100 ml/min (standard conditions). The results are shown in Figure 7.
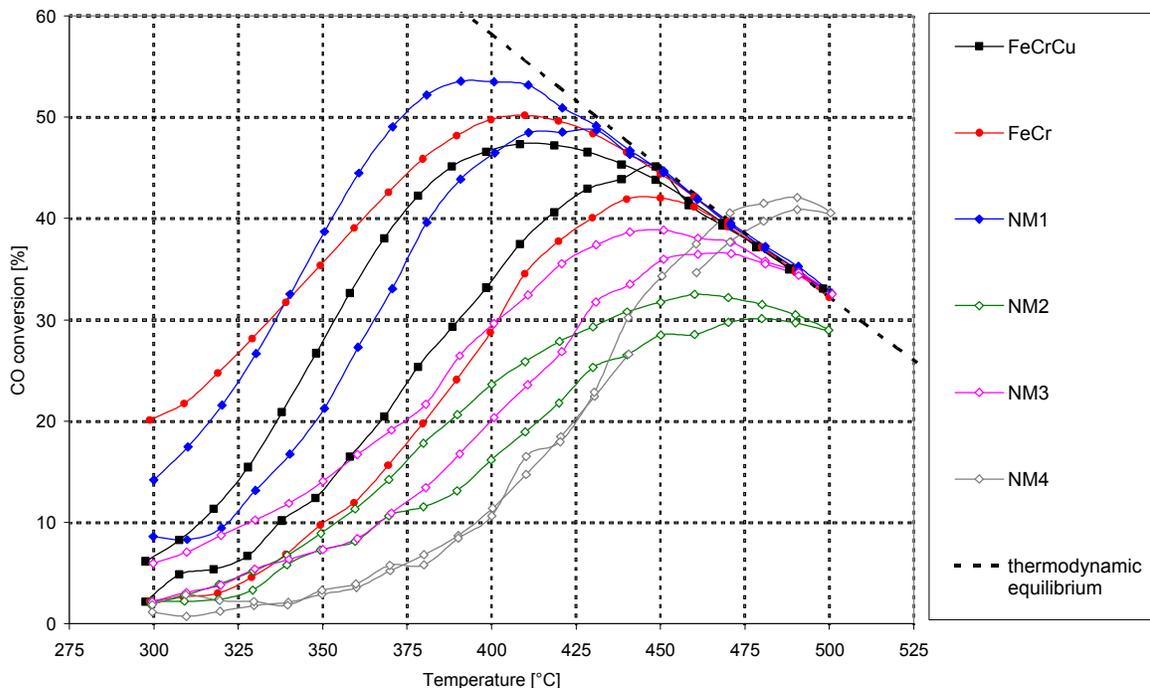
Figure 7 Activity measurement 300–500°C, atmospheric pressure, 212-300μm sieve fraction

All catalysts appear to deactivate during the experiment. As expected the Fe_2O_3/Cr_2O_3-based catalysts have a similar activity, especially in the second part of the experiment during cooling. For the noble metal catalysts, the spread is significant with NM1 being the most active and NM4 the least active. NM4 shows a tendency for catalysing methanation, with methane being detected at temperatures as low as 320°C.

Activity measurements at elevated pressure
Using the parallel flow setup that is available, activity measurements could be done at a maximum pressure of 2 barg. Currently, adjustments are being made to go up to 5 barg.

Stability measurements in absence of H_2S
Stability of the catalysts is tested for 200 hours. The main part of the experiment runs at 450°C (equilibrium conversion: 45%), while the temperature is reduced periodically to 400°C (equilibrium conversion: 58%) to assess the activity of the catalyst further from thermodynamic equilibrium. The noble metal catalysts have been diluted as powders with 95% of high purity α-Al_2O_3, to a total of 100 mg of sieve fraction. The Fe_2O_3/Cr_2O_3-based catalysts have been tested as 200 mg of undiluted sieve fraction. Both types of sample were tested with a total flow of 100 ml/min (standard conditions). The results for the full 200 hours are shown in Figure 8.
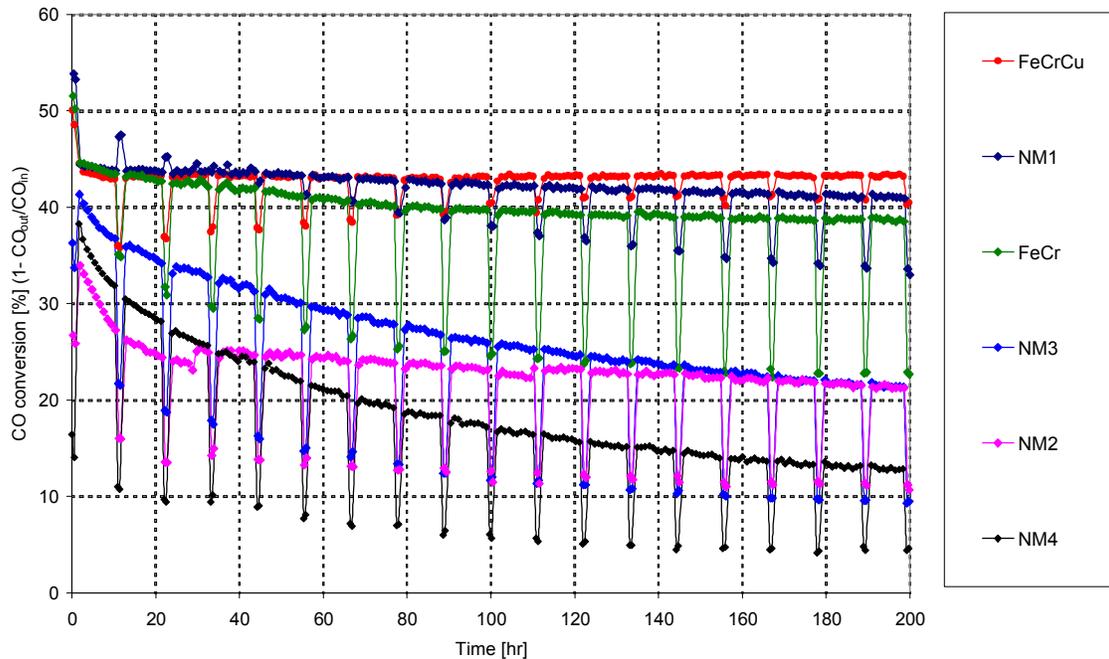
Figure 8 Stability measurement 450°C (400°C), atmospheric pressure, 212-300μm sieve fraction

Clearly, most catalysts deactivate over the first 130 hours of measurement. In contrast the FeCrCu catalyst appears to gain activity. Up till now only the NM2 catalyst appears to stabilise. The noble metal based catalysts clearly perform worse than the Fe_2O_3/Cr_2O_3-based ones except NM1. FeCrCu and NM1 approach equilibrium conversion.

Stability measurements in the presence of H_2S
Presently, stability measurements with the addition of 20 ppmv of H_2S are about to start.

Progress
- Structure, composition and decomposition pathway of Mg-Al Hydrotalcite have been determined. The Mg/Al ratio is found to be 1.8, the net formula is $Mg_{0.64}Al_{0.36}(OH)_2(CO_3)_{0.18}$·1.0 H_2O. At about 250°C the carbonate-water layer disappears and with this the presupposed transport path for CO_2 through a dense hydrotalcite membrane.
- Porous hydrotalcite based membranes where the transport and selectivity are based on affinity of CO_2 to the micro pore walls are now the focus of R&D at ECN. Coatings based on nano particulate dispersions or sol gel synthesized particles and sensitizing microporous supports are the three routes to achieve our goal.
- WGS catalysts have been found that are stable at least for 200 h under experimental conditions without sulphur compounds.
- More detailed system analyses show that doubling the target permeation (derived from H_2-selective WGS-MR) of CO_2 selective WGS-MR already leads to a competitive advanced membrane reactor configuration. Moreover, the high CO_2 purity as well as the flexibility with respect to syngas conversion and CO_2 separation in combination with the reduced hydrogen loss due to combustion with

traces of oxygen are important advantages for implementation of the CO_2-selective WGS-MR

- ECN has supported characterization work connected with the GCEP R&D at the TU Delft. Permporometry and physisorption measurements have successfully been performed on micro porous substrates to be modified with Atomic Layer Deposition in a later stage.

Future Plans

Task 1 The present results on system evaluations will be combined with the results on reactor design (Task 5) in the previous Annual report and published in a peer reviewed scientific journal. Further activities are on hold until more progress is made with the membrane materials development.

Task 3 This task will be the main focus of the project for the coming period. Emphasis will be on the synthesis and characterization of nano sized particles either using sol-gel chemistry or micro emulsion based techniques. These particles will first be coated on well defined smooth surfaces and characterized. The next step will be coating on a porous support and characterization of membrane performance as to selectivity and flux. Recently a DoE report [5] has become available to the public that also deals with the development of hydrotalcite based CO_2 selective membranes. The findings therein will be used to our advantage and may considerably speed up our developments.

Task 4 Catalyst screening will be pursued as described (effect of sulphur poisoning and higher pressure). Only for the best candidate kinetics will be determined.

Task 5 See Task 1.

Publications

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8. Haije, W.G., Materials science as a basis for future technologies for CO_2 Capture and storage and H_2 storage. Presentation at the spring meeting of the Royal Dutch Chemical Society (KNCV), Ede, April 2007:

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