Chemical-Looping with Oxygen Uncoupling using combined Mn-Fe oxides, testing in batch fluidized bed

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Abstract

Chemical-looping with oxygen uncoupling (CLOU) has been studied with gaseous and solid fuel in a small fluidized bed batch reactor, using new Fe-Mn-based oxygen carriers. CLOU is a development of chemical-looping combustion, using oxygen carriers with the ability to release oxygen, which can react directly with the fuel. The carbon dioxide from the combustion is inherently obtained as separated from the rest of the flue gases. In this work manganese is combined with iron oxides, giving new bimetallic oxide compounds with different thermodynamic properties compared to pure manganese oxides. Four different combinations of iron manganese oxide have been examined by decomposition in N2 and also reaction with methane and syn-gas. F3, a material with a molar ratio of Fe:Mn of 2:1, showed the best behaviour in terms of its release of oxygen, take up of oxygen, fluidizability and methane conversion. Therefore, F3 was also tested with solid fuel using inert fluidization gas, N2. The char particles effectively remove oxygen released as it is converted to CO2. Thus, CO2 will represent the oxygen release. The tests show that the particles release oxygen corresponding to approximately 0.5% of their mass. Moreover, a test where steam was added in the fluidization gas showed high gas conversion, with essentially no unconverted gas. Thus, the tests indicate that the F3 particles, if used in chemical-looping of solid fuels, could contribute both to faster fuel conversion and to higher conversion of gas, as compared to a normal oxygen carrier that does not release oxygen.

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1. Introduction:

One of the technologies that can be used for CO2 capture is chemical looping combustion (CLC). One of the most important benefits of this technique is that CO2 and H2O are separated from other components of flue gases.

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like excess O$_2$ and N$_2$ as a part of the process. By eliminating the need for separation of gases costly and energy consuming equipment is avoided [1].

The CLC system is composed of two fluidized bed reactors. One of them is an air reactor where an oxygen carrier, usually a metal oxide, is oxidized by air. The oxygen carrier will then be transported to the second reactor, the fuel reactor. In the fuel reactor the added fuel reacts with the oxygen carrier to produce CO$_2$ and H$_2$O. The reduced oxygen carrier is then again transported back to air reactor to be re-oxidized back to its original state. The total amount of heat released from fuel reactor and air reactor is equal to the heat released from ordinary combustion [1]. The flue gases from the fuel reactor consist almost only of CO$_2$ and H$_2$O. The H$_2$O can be condensed and pure CO$_2$ can be compressed and transported to an appropriate storage location. The flue gases from the air reactor mainly consist of nitrogen and a small amount of oxygen which can be released to the atmosphere. Since CO$_2$ is inherently separated from the nitrogen and oxygen in the flue gas, there is no direct energy penalty for the gas separation [1].

The majority of the published work on CLC has focused on gaseous fuels. Solid fuels, like coal, are more abundant and cheaper than gaseous fuel. Consequently, it would be more beneficial if the CLC process could be adapted to solid fuels. However, metal oxide and solid fuel do not react directly, but only via gaseous intermediates [2]. Therefore, in order to increase the reaction rate, fluidizing the mixture of fuel and particles in the fuel reactor with H$_2$O and/or CO$_2$ is proposed. The solid fuel will be gasified by H$_2$O or CO$_2$ to H$_2$ and CO that can react with the oxygen carrier [3].

Another option is Chemical-Looping with Oxygen Uncoupling (CLOU) which is an improvement of chemical looping combustion [4]. In this method the solid fuel is oxidized through 2 steps. Firstly, oxygen can be released by the oxygen carrier through the following reaction:

$$\text{Me}_x\text{O}_y \leftrightarrow \text{Me}_{x-2}\text{O}_y + \text{O}_2 (g) \quad (1)$$

Secondly, the fuel reacts with gas-phase oxygen, like in normal combustion, and produces CO$_2$ and H$_2$O according to reaction 2.

$$\text{C}_n\text{H}_{2m} + (n + m/2) \text{O}_2 (g) \leftrightarrow n\text{CO}_2 + m\text{H}_2\text{O} \quad (2)$$

The oxygen carrier will then be transported to the air reactor to be oxidized with air just as in CLC.

$$\text{O}_2 + \text{Me}_{x-2}\text{O}_y \leftrightarrow \text{Me}_x\text{O}_y \quad (3)$$

The total amount of heat released from the reactions above is the same as for conventional combustion and as for normal CLC. The main benefit with CLOU, as compared to CLC, is that the slow gasification of the solid fuel is eliminated [4]. The trickiest part of CLOU is selecting an appropriate oxygen carrier. Oxygen carriers for CLOU should have special characteristics and also react reversibly at elevated temperature. The oxygen carrier must be oxidized with oxygen in air reactor and also release gaseous O$_2$ in the fuel reactor at appropriate temperature and oxygen partial pressures [4]. Three metal oxides of manganese, copper, cobalt have appropriate equilibrium pressure of gaseous oxygen at 800-1200 °C which is the desired temperature interval in CLOU [4].

It should be noted that oxides with CLOU properties may react as both conventional CLC materials and CLOU materials. When such materials react with gaseous fuel, it is difficult to distinguish between the mechanisms.

2. Thermodynamics:

As discussed above, the oxygen carrier in CLOU needs special thermodynamic properties to react reversibly at elevated temperatures. Thermal analysis of three metal oxides system has been performed by Mattisson et al. [4], for instance Mn$_2$O$_3$/Mn$_3$O$_4$. This material releases oxygen in the gas phase through the following reversible reaction:

$$6\text{Mn}_2\text{O}_3 \leftrightarrow 4\text{Mn}_3\text{O}_4 + \text{O}_2 (g) \quad \Delta H_{850}^{\circ} = 193.9 \text{ kJ/mol } \text{O}_2 \quad (4)$$

For Mn$_2$O$_3$/Mn$_3$O$_4$ the temperature in which the equilibrium pressure of gas –phase O$_2$ is equal to partial pressure of O$_2$ in air is 899 °C, which means that Mn$_2$O$_3$ releases oxygen in air at temperature above 899 °C and Mn$_3$O$_4$ takes up oxygen at lower temperature in air [4]. The oxidized particle will be transported to the fuel reactor in which the
partial pressure of O₂ is lower. This lack of oxygen in the fuel reactor causes the oxygen carriers to decompose and release gaseous O₂. The amount of oxygen released and the maximum concentration of oxygen are dependent on the fuel reactor temperature. The fuel reactor temperature is influenced by the temperature of the incoming particles, the circulation rate and heat of reaction in the fuel reactor. For Mn₂O₃/Mn₃O₄ the overall reaction in the fuel reactor is exothermic, which results in a temperature increase in the fuel reactor and consequently the oxygen carrier will release more gaseous oxygen. A higher partial pressure of oxygen will improve the overall conversion rate of solid fuel [4].

Applying pure manganese oxide in CLOU has some drawbacks because the relevant equilibrium concentrations applicable for CLOU mean operations at relatively low temperatures. For example, to have 5% for the maximum outlet partial pressure of O₂ from air reactor, the maximum temperature for Mn₂O₃ corresponding to this partial pressure is 820°C[4]. For solving these thermodynamic limitations combination of active manganese oxide with other active or inert material can be applied. Iron, nickel and silicon are materials that can be combined with manganese oxides to improve its characteristics [4-5]. In this work different combinations of iron and manganese were studied in a fluidized batch reactor to investigate their oxygen release and uptake potentials and also their reactivity with respect to solid fuels and methane. Iron manganese oxide has different properties from pure manganese oxide and is possible to oxidize by 5% O₂ at temperatures higher than 820°C.

3. Experimental:

**Materials.** The oxygen carriers studied in this work are particles with different molar ratios of Fe/Mn which were produced using spray drying at VITO in Belgium. Oxygen carrier particles with high sphericity, good free-flowing properties and homogeneity on the micro-scale, were prepared by the industrial spray-drying method, using different commercial raw materials. Powder mixtures of α-Mn₂O₃ and α-Fe₂O₃ were dispersed in deionized water containing organic additives. The water-based suspension was continuously stirred with a propeller blade mixer while being pumped to the 2-fluid spray-dry nozzle, positioned in the lower cone part of the spray-drier. After spray-drying, the fraction between 106 and 212 mm was separated from the rest of the spray-dried product, by sieving the chamber fraction. In order to obtain oxygen carrier particles with sufficient mechanical strength, sintering was performed in air at 1100 °C, for 4 h. After sintering, the particles were sieved again; hence all particles in the tests were of well-defined sizes[6]. Table 1 shows specifications of the different particles used in this work. The size of the particles used is in the range 125-180 μm. The solid fuels which were used for these experiments are Petroleum coke and Colombian coal. All fuels were crushed and sieved to achieve particles in the size range 125-180 μm. Table 2 presents the fuel analysis.

**Table 1- Composition of iron manganese oxide as oxygen carrier**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Molar ratio(Fe:Mn)</th>
<th>Mass ratio(Fe₂O₃:Mn₃O₄)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>4:1</td>
<td>80.7:19.3</td>
</tr>
<tr>
<td>F3</td>
<td>2:1</td>
<td>67.7:32.3</td>
</tr>
<tr>
<td>F5</td>
<td>1:2</td>
<td>34.4:65.6</td>
</tr>
<tr>
<td>F7</td>
<td>1:4</td>
<td>20.7:79.3</td>
</tr>
</tbody>
</table>

**Table 2- Fuel analysis**

<table>
<thead>
<tr>
<th>Fuel</th>
<th>Hi[MJ/kg] (as received)</th>
<th>Proximate [wt%, as received]</th>
<th>Ultimate [wt%,d.a.f]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>M</td>
<td>A</td>
<td>Combustibles</td>
</tr>
<tr>
<td>Colombian coal</td>
<td>25.9</td>
<td>6.2</td>
<td>11.9</td>
</tr>
<tr>
<td>Petroleum coke</td>
<td>30.9</td>
<td>8</td>
<td>0.5</td>
</tr>
</tbody>
</table>

**Experimental procedure.** The CLOU property of iron manganese oxide is examined by decomposition in N₂ and moreover the reaction with both solid and gaseous fuel was examined. For the investigation of the oxygen release and reactivity of gaseous fuel, the experiments were done in a fluidized bed quartz reactor which has length of 820 mm and with a porous quartz plate of 30 mm in diameter placed 370 mm from the bottom. A sample of 15 g
of oxygen carriers with size of 125-180 μm was placed at the porous plate and the reactor was then heated to the temperature of interest in a flow of 900 mL\textsubscript{min}/min containing 5% O\textsubscript{2} in N\textsubscript{2} in order to prevent uncontrolled release of oxygen and to ensure that the oxygen carriers are adequately oxidized prior to the experiments. As the required conditions were reached, the particles were fluidized by 600 mL\textsubscript{min}/min of pure N\textsubscript{2}, and the outlet oxygen concentration was measured during the inert period. The particles were exposed to consecutive cycles of oxidizing and inert periods at a temperature of 900°C and also cycles in which the temperature for oxidation was still 900°C but the temperature was raised to 1000°C during the inert period. For reactivity evaluation, the particles were exposed to CH\textsubscript{4} and syngas. The reactor was purged from reactive gases and gaseous products by introduction of N\textsubscript{2}, so the oxidation and the reduction steps were separated by an inert step. Table 3 presents detailed plan of experiments. This plan was carried out for all samples.

Table 3- Experimental plan for decomposition and gaseous fuel. \( F_x \) is flow in period \( x \), i.e. Ox(idation), Red(uction) and In(ert)

<table>
<thead>
<tr>
<th>No of cycles</th>
<th>Reducing gas</th>
<th>( F_{\text{Ox}} ) (mL\textsubscript{min}/min)</th>
<th>( F_{\text{In}} ) (mL\textsubscript{min}/min)</th>
<th>( t_{\text{In}} ) (s)</th>
<th>( F_{\text{Red}} ) (mL\textsubscript{min}/min)</th>
<th>( t_{\text{Red}} ) (s)</th>
<th>( T_{\text{Ox}} ) (°C)</th>
<th>( T_{\text{Red}} ) (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>nitrogen</td>
<td>900</td>
<td>600</td>
<td>360</td>
<td>-</td>
<td>-</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>1</td>
<td>nitrogen</td>
<td>900</td>
<td>600</td>
<td>360</td>
<td>-</td>
<td>-</td>
<td>900</td>
<td>900 → 1000</td>
</tr>
<tr>
<td>3</td>
<td>methane</td>
<td>900</td>
<td>600</td>
<td>60</td>
<td>450</td>
<td>20</td>
<td>950</td>
<td>950</td>
</tr>
<tr>
<td>3</td>
<td>syngas</td>
<td>900</td>
<td>600</td>
<td>60</td>
<td>450</td>
<td>80</td>
<td>950</td>
<td>950</td>
</tr>
<tr>
<td>3</td>
<td>nitrogen</td>
<td>900</td>
<td>600</td>
<td>360</td>
<td>-</td>
<td>-</td>
<td>900</td>
<td>900</td>
</tr>
<tr>
<td>3</td>
<td>nitrogen</td>
<td>900</td>
<td>600</td>
<td>360</td>
<td>-</td>
<td>-</td>
<td>900</td>
<td>900 → 1000</td>
</tr>
</tbody>
</table>

For solid fuel, the experiments were performed in a fluidized-bed reactor of quartz. The reactor is the same as the reactor which had been used previously by Leion et al. [7]. A sample of 20 g of F3 particles with a size of 125-180 μm was placed at the porous plate and heated to the temperature of interest in a flow of 1000 mL\textsubscript{min}/min containing 5% O\textsubscript{2} in N\textsubscript{2}. The particles were then alternatingly exposed to an oxidizing gas of 5% O\textsubscript{2} in nitrogen and a reducing period where 0.1 g of fuel was introduced to the bed of oxygen carriers which was fluidized with 900 mL\textsubscript{min}/min of pure N\textsubscript{2}. In one additional test the flow during the reduction phase was 450 mL\textsubscript{min}/min of N\textsubscript{2} and 450 mL\textsubscript{min}/min of steam. 450 mL/min of inert sweep gas was introduced to the system at the top of the reactor together with the solid fuel throughout the reducing period to ensure that the pulverized fuel did not get stuck in the feed. However this sweep gas did not enter the hot reaction zone of the reactor. The oxidation and the reduction steps were separated by an inert step with 900 mL\textsubscript{min}/min of pure N\textsubscript{2}. The reactivity of F3 particles was investigated with two solid fuels: Colombian coal and petroleum coke. For both fuels the temperature was 950°C and also pure inert was used as fluidizing flow in reduction. Petroleum coke was also examined at a temperature of 900°C and 1000°C and in the additional test where steam was used with N\textsubscript{2}. The solid fuels experiments were carried out 3-5 cycles for each experimental set up.

The gas from the reactor was led to an electric cooler for removing water and then to a Rosemount NGA 2000 Multi-Component gas analyzer, giving the concentrations of CO, CO\textsubscript{2}, CH\textsubscript{4} and O\textsubscript{2} and also the gas flow. The temperature was measured 5 mm under and 10 mm above the porous quartz plate using Pentronic CrAl/NiAl thermocouples with inconel-600 enclosed in quartz shells. The exothermic nature of both the oxidation and reduction means that the bed temperature varied somewhat during the cycle. The temperature presented in the paper is the set-point temperature, i.e. the temperature at the beginning of the reduction. From high frequency measurements of the pressure drop it was possible to see if the bed was fluidized.

**Data Evaluation.** The degree of conversion, \( X \), describes the extent to which the oxygen carriers are oxidized and is defined as follows:

\[
X = \frac{m - m_{\text{red}}}{m_{\text{ox}} - m_{\text{red}}} \tag{5}
\]

where \( m \) is the actual mass of the sample, \( m_{\text{ox}} \) is the mass of the fully oxidized sample, and \( m_{\text{red}} \) is the mass of the sample in its fully reduced form. The degree of oxidation of oxygen carriers during reduction with methane as a function of time is calculated from the outlet gas concentrations using eq 6.
\[
X_i = X_{i-1} - \frac{1}{t_1 - t_0} \left( \frac{4n_{\text{out}}(p_{\text{CO}_2,\text{out}} + 3p_{\text{CO},\text{out}} - p_{\text{H}_2,\text{out}})}{M_0P_{\text{tot}}} \right) dt
\]

Correspondingly, the degree of conversion for the oxidation period is determined using the relationship

\[
X_i = X_{i-1} - \frac{1}{t_1 - t_0} \left( n_{\text{in}}(p_{O_2,\text{in}} - n_{\text{out}}p_{O_2,\text{out}}) \right) dt
\]

where \( X_i \) is the conversion as a function of time for a period \( i \), \( X_{i-1} \) is the degree of conversion after the foregoing period; \( t_0 \) and \( t_1 \) are respectively the times for the start and the finish of the period; \( M_0 \) is the molar mass of active oxygen in the unreacted oxygen carrier; \( n_{\text{in}} \) and \( n_{\text{out}} \) are the molar flows of dry gas entering and exiting the reactor, respectively; \( P_{\text{tot}} \) is the total pressure; \( p_{\text{CO}_2,\text{out}} \), \( p_{\text{H}_2,\text{out}} \), and \( p_{\text{CO},\text{out}} \) are the outlet partial pressures of \( \text{CO}_2 \), \( \text{H}_2 \), and \( \text{CO} \) after removal of water vapour; and \( p_{O_2,\text{in}} \) and \( p_{O_2,\text{out}} \) are the partial pressures of incoming and exiting oxygen.

In order to be able to compare oxygen carrier materials which contain different amounts of oxygen, a mass-based conversion, \( \omega \), is defined as follows:

\[
\omega = \frac{m_{\text{CO}_2}}{m_{\text{ox}}} = 1 + R_0(1 - X)
\]

For analysis of gas conversion, the fraction of \( \text{CO}_2 \) in the outlet gas flow was calculated on dry basis as follows:

\[
\gamma_{\text{red}} = \gamma_{\text{CO}_2} = \frac{p_{\text{CO}_2}}{p_{\text{CH}_4} + p_{\text{CO}_2} + p_{\text{CO}}}
\]

### 4. Results and Discussions:

**Gaseous Fuel.** Figure 1 shows the oxygen concentration during an inert phase at a temperature of 900°C for different oxygen carrier particles. As seen F3 and F1 release oxygen whereas F7 and F5 release no oxygen. Moreover there is no change in behaviour of F7 and F5 during temperature change in the inert phase. Problems in fluidization for F7 and F5 were noted. F7 was sensitive to higher temperature and when temperature was increased to 950°C and 1000°C the fluidization became problematic. In the case of F5, the fluidization became unstable after a few cycles and during some cycles it was totally defluidized. For some cycles of inert, the temperature was raised from 900°C to 1000°C to investigate the release of oxygen during temperature increase. The results are presented in figure 2 for both F1 and F3. For the first cycle with temperature increase which is before the methane cycles, the temperature increase leads to a significant amount of oxygen release for both particles, especially for F3. In comparison, the oxygen release for the inert cycle following the syn-gas cycles also results in a release of oxygen, although smaller especially for F3, see last cycle in figure 2. Figure 2 shows that the release of oxygen for F3 is higher than F1. Some difficulties in fluidization of F1 were also observed.

Figure 3 demonstrates the outlet dry gas concentration for reduction and regeneration of F3 oxygen carrier particles at 950°C using methane as fuel. In figure 3 (left) there is a \( \text{CO}_2 \) peak and also high \( \text{CH}_4 \) peak which suggests that the F3 particles cannot convert all methane to \( \text{CO}_2 \). In the figure a small peak of \( \text{CO} \) also can be seen. From the figure 3 (right) a temperature increase during oxidation can be seen as a result of exothermic oxidation. All the other three particles show a similar type of behaviour although the amount of \( \text{CO}_2 \) and the take up of oxygen vary for each particle.
Figure 3 shows the gas conversion, from equation 9, as a function of mass-based conversion at 950°C with methane. The methane conversion for F3 oxygen carrier is higher than the others. The value for $\gamma$ does not start at 1 because it decreases slightly due to release of oxygen during the short inert period before the reduction.

It should be mentioned that all the particles showed a full conversion of syngas. Of these four particles, F3 showed the best CLOU behaviour in terms of its release of oxygen, take up of oxygen, fluidizability and methane conversion. Therefore, it was also tested with solid fuel. Moreover it has been tested in circulating fluidized bed reactor by Rydén et al. [8].

Figure 4 shows the gas conversion, $\gamma$, vs. the mass-based conversion, $\omega$, at 950°C using methane.

Solid fuel. In figure 5 the corrected outlet gas concentrations, i.e. concentration before adding the sweep gas, are shown as a function of time for the reducing period and the following oxidation period for F3 particles and Colombian coal at 950 °C. The oxygen concentration at the end of oxidation is 5% and when the fluidizing gas is switched to nitrogen it starts to fall down to around 1% because of oxygen release. When the fuel is introduced to the reactor a small peak of CH$_4$ and CO can be seen in the beginning of the reaction due to devolatilization of the fuel. Some volatiles react with the oxygen carrier and CO$_2$ increases rapidly. The peak value for CO$_2$ is lower than the peak for CH$_4$ and CO. The oxygen concentration falls to zero as the fuel conversion is much faster than oxygen release. After devolatilization is finished, the remaining char can only be converted by reaction with oxygen released from the oxygen carrier. This is because the fluidizing gas is nitrogen, so there is no gasification. Thus, the CO$_2$ is a measure of oxygen release. After switching to oxidizing gas the level of oxygen increases and this is accompanied by some small increase in temperature as a result of exothermic oxidation. There is a CO$_2$ peak during oxidation which shows that there is some fuel left in the bed which has not been converted.
Figure 6 shows similarly the gas concentrations for F3 particles and petroleum coke. The gas concentrations and temperature profile are qualitatively similar to the previous solid fuel. Lower initial peaks of CH4, CO and CO2 are a result of lower volatiles content of the petroleum coke. The reducing period for petroleum coke is also longer.

Petroleum coke at temperature 900 and 1000 °C also shows a similar type of behaviour as the one shown in Figure 6, although the rate of reaction and the flue gas concentrations are different. Figure 7 shows the corrected CO2 concentration, i.e. the concentration before addition of sweep gas, as a function of mass-based conversion during reduction with petroleum coke at 900, 950, 1000°C and also using steam at 950°C. The change in mass-based conversion suggests that the oxygen-carrier particles are able to release oxygen corresponding to approximately 0.5% of their mass. The concentration of CO2, which corresponds to the oxygen release rate when F3 and fuel is fluidized by nitrogen, is increased by temperature increase. Adding steam as fluidizing gas in the reduction period, see Figure 7, increases the CO2 concentration as the char can also be gasified by steam. Thus, a combination CLC and CLOU, in other words of steam gasification with syn-gas being oxidized by the oxygen carrier, and combustion through oxygen release, determines the fuel conversion rate. Moreover, in Figure 8 it can be seen that after devolatilization the CO concentration falls to zero which indicates full conversion the gas, in contrast to experiments with ordinary oxygen carriers without release of gaseous oxygen, where there is a significant fraction of unconverted CO.
5. Conclusions:

Four combinations of iron manganese oxide were examined by decomposition in N\textsubscript{2} and also reaction with methane and syngas. The F3 particles, with a molar ratio of Mn:Fe of 2:1, showed the best CLOU behaviour in terms of its release of oxygen, take up of oxygen, fluidizability and methane conversion. Therefore, F3 was also tested with solid fuel. In these tests oxygen released is consumed by the char particles forming CO\textsubscript{2}, and with an inert fluidizing gas, i.e. N\textsubscript{2}, gasification of char is negligible. Thus, for the char conversion phase following the initial gasification the CO\textsubscript{2} concentration will represent the oxygen release. The tests indicate that the oxygen carrier releases oxygen, corresponding to around 0.5% of its mass or more. Although this release is small, e.g. compared to copper-based CLOU materials, it is nevertheless sufficient to be of interest for application to solid fuels. Firstly, the release of oxygen may contribute to an increased char conversion rate, i.e. compared to that of steam gasification only. Secondly, it may give very high conversion of the gas, as illustrated above in the tests with steam added.

6. Acknowledgment:

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7. References: