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Dedicated to Professor Liviu Literat
On the occasion of his 85th birthday

APPLICATIONS OF CHEMICAL LOOPING COMBUSTION TO ENERGY CONVERSION PROCESSES

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ABSTRACT. Among various carbon capture technologies, chemical looping combustion (CLC) is a promising option to reduce both energy and cost penalties for CO_2 capture. This technique uses an oxygen carrier (usually a metalic oxide) for fuel oxidation followed by reoxidation of reduced oxygen carrier by air and/or steam. Both fuel and air reactors are operated in fluidised conditions. This paper investigates the optimisation of process operation conditions for two CLC cases, one based on natural gas and one based on syngas as fuels. The investigated CLC processes using various metallic oxides (e.g. iron, nickel, copper and mangan oxides) as oxygen carriers are aiming to find optimum process conditions (pressure, temperature, oxygen carrier flow rate, molar ratio among reactants, oxygen carrier concentration in the solid phase etc.) for an almost complete fuel conversion as well as for total decarbonisation of the used fuel.

Keywords: *Energy conversion with carbon capture, Chemical looping combustion (CLC), Oxygen carriers, Optimisation of process operation conditions.*

INTRODUCTION

In the last decade, significant progress has been made towards a better understanding of the world climate and of the long-term impact of climate change. There is now evidence that the mean annual temperature at the earth's surface increased over the past 200 years. This temperature increase is commonly known as global warming. It has been established that emission of greenhouse gases (CO_2, NO_x, SO_x, CH_4) are the main contributor to global warming, and CO_2 is the most prevalent of these gas emissions. Statistics indicate that the CO_2 emissions resulting from human activity have led to an increase in the atmospheric CO_2 concentration, from a pre-industrial level of 280 to 380 ppm [1].

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There are several ways in which to reduce anthropogenic CO_2 emissions: increase the use of renewable sources of potential energy, such as hydro-, wind and solar power; promote CO_2 uptake in biomass; switch from fossil fuels to biomass; expand the nuclear power sector; and finally, reduce global energy intensity. However, it is likely that society will remain highly dependent on fossil fuels for some time to come [2]. Hence an additional alternative to reduce the net emissions of CO_2 has been suggested: combining the use of fossil fuels with CO_2 capture and storage (CCS), which would yield to low carbon sources of heat and power [2].

A number of known techniques can be used to carry out this separation, but a major disadvantage with most of these techniques is the large amount of energy that is required to obtain CO_2 in pure form, which means that the efficiency of power plants decrease with about 8–12 net electricity percentage points [2]. Combustion of fossil fuels (coal, lignite, oil, natural gas) release a massive amount of carbon as carbon dioxide into the atmosphere. It is estimated that fossil fuel-based power generation contributes today to about one-third of the total carbon dioxide released.

In order to capture CO_2, there are currently a number of available processes as follows: (i) pre-combustion, in which the fuel is decarbonized prior to combustion, (ii) oxy-fuel combustion, which uses pure oxygen obtained from cryogenic air separation and (iii) post-combustion separation, which separate CO_2 form the flue gases using different approaches. These techniques are energy intensive, resulting in a significant decrease of the overall energy conversion efficiency and as a result in a price increase of the produced electricity. For instance, gas-liquid absorption using alkanolamines (e.g. mono-etnahol-amine - MEA, methyl-diethanol-amine - MDEA) which are the most technologically and commpercially mature CO_2 capture processes has as major negative impact the high heat duty needed for solvent regeneration (in the range of 3 to 4 MJ/kg CO_2). Considering all these factors, chemical-looping combustion (CLC) appears to have the potential for delivering a most efficient and low cost technology. This process was initially proposed to increase thermal efficiency in power generation stations, but later on was identified as having inherent advantages for CO_2 separation with minimum energy losses [1].

The chemical looping combustion is implying the usage of several interconected circulated bed reactors, as presented in Figure 1 (for the case of gaseous fuels like natural gas or syngas resulted from catalytic reforming / gasification processes). The fuel (either gaseous, liquid and solid) is introduced to the fuel reactor where it reacts with an oxygen carrier (OC) to convert the fuel to CO_2 and H_2O. The gas phase resulted from the fuel reactor is cooled, water condense separated and CO_2 stream is ready for storage (inherent CO_2 capture is one of the most atractive feature of chemical looping systems). The reduced stage of the solid carrier is then pneumatically transported to a separate reactor where it is oxidized back to its original state. The reoxidation process can be made by air, steam or o combination of both. The energy integration between fuel and air reactors is a key aspect of this technology (the reactions in the fuel reactor are mainly endothermic and the oxygen carrier reoxidation is always exotermic), solid flow being used as heat transport media [3-4].

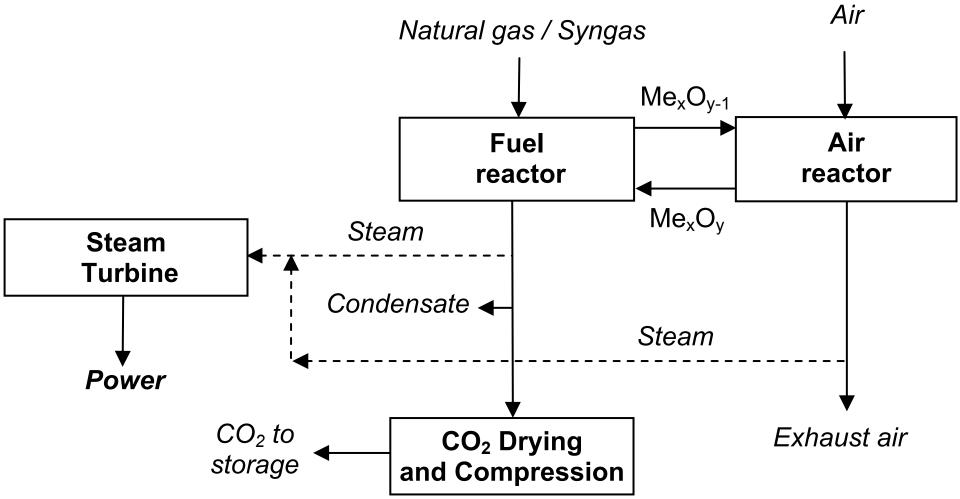

Figure 1. Chemical looping combustion (CLC) of gaseous fuels

In the fuel reactor, the oxygen carrier is reduced by the fuel to lower oxidation stage. This paper is investigating gaseous fuels like natural gas or syngas [5]. The fuel (hydrocarbon) oxidation reaction is the following:

$$(2n+m)Me_xO_y + C_nH_{2m} \rightarrow (2n+m)Me_xO_{y-1} + nCO_2 + mH_2O \qquad (1)$$

Me_xO_y is a metal oxide and Me_xO_{y-1} represents its reduced form.

The reduced form of the oxygen carrier is pneumatically transported to a separate reactor where it is regenerated (reoxidation) by air according to the reaction 2. Reoxidation can be done also with steam.

$$Me_xO_{y-1} + \frac{1}{2} O_2 \rightarrow Me_xO_y \qquad (2)$$

The reoxidised oxygen-carrier is then closing the cycle being transported back to the fuel reactor. The reduction process in the fuel reactor (reaction 1) can be either endothermic or exothermic, depending on the oxygen carrier and the fuel, while the oxidation process in the air reactor (reaction 2) is exothermic [5-6]. In theory, the oxygen carrier can be used indefinetly. However, the solid material must be renewed by a makeup flow of new material due to undesirable attrition / fragmentation or reactivity loss [6]. The usage of an inert support for the oxygen carrier is significantly improve the mechanical strength.

In chemical-looping combustion (CLC), a metal oxide is used as an oxygen carrier which transfers oxygen from the combustion air to the fuel [7]. The main advantage with CLC compared to normal combustion is that CO_2 is inherently separated from the other flue gas components, i.e. N_2 and unused O_2 and thus, costly equipment and efficiency losses for separation of CO_2 are avoided [8]. Although the advance of the CLC technology involved works carried out at different research levels, continuous operation in a CLC prototype is necessary to demonstrate the validity of this technology and to verify the usefulness of the particles developed. The CLC process was first successfully demonstrated by Lyngfelt et al. at Chalmers University, Sweden, in a 10 kWh prototype during 100 h of continuous operation burning natural gas and using nickel based oxygen carrier particles [9].

Most research to date on CLC is mainly concentrated on gaseous fuel, and CLC applications with solid fuels, e.g. coal, biomass, are still limited [10]. In the development of suitable oxygen carrier material it is important to test a significant number of materials with varying metal oxide/support combinations and production conditions [8]. OC was the basis for CLC and acted as oxygen conductor as well as energy carrier in CLC, which is composed of the active metal oxides and inert support. Until now, Fe_2O_3, CuO and NiO are widely used in CLC as the active oxigen carriers, and a variety of inert materials have been reported as the inert supports for OC, such as Al_2O_3, SiO_2, ZrO_2, etc. among which, Al_2O_3 is recognized as one of the most promising supports [10].

A number of materials have been identified as suitable OC in a CLC system: metal oxides based on iron, nickel, manganese, copper or cobalt, supported on alumina, zirconia, silica, titania, or bentonite. An OC must withstand a high number of redox cycles, both chemical and physically, in order to be used in a CLC process. Thermodynamic characteristics of the metalic oxides vs. temperature is an important aspect, Ellingham diagrams being used. In addition, other characteristics such as high oxygen transport capacity, complete fuel conversion to CO_2 and H_2O, negligible carbon deposition, good properties for fluidization (no presence of agglomeration) and easy preparation to reduce costs are also important [7].

There are some important criteria for the selection of oxygen-carrier particles. The basic requirements for OC are mechanical stability, lowest possible costs and high oxygen transport capacity [11]. The thermodynamic equilibrium for the reaction with the fuel has to be favorable in order to achieve high fuel conversion to CO_2 and H_2O. Metal oxide systems which are feasible for use as oxygen carriers in CLC are Mn_3O_4/MnO, Fe_2O_3 / Fe_3O_4, NiO / Ni, CuO / Cu and CoO / Co. Also the rate of oxidation and reduction has to be sufficiently fast. Otherwise the amount of oxygen-carrier needed in the reactors would be too large. Moreover the oxygen transfer capacity needs to be sufficient. Since the proposed reactor system consists of fluidized beds, the particles need to have a low tendency for fragmentation and attrition. It is also vital that they do not agglomerate under real reaction conditions [9]. In view of maintaining a sufficient fuel reactor temperature for a high conversion rate, CuO has been highly appraised and intensively studied for the exothermic characteristics of its reduction reaction with various fuels [7]. In a CLC system, the solid circulation between the reactors has to be sufficient to transport the adequate amount of oxygen for fuel combustion, and to transfer sufficient sensible heat from the air reactor to the fuel reactor for those oxide systems where the reaction in the fuel reactor is endothermic [9].

In the manufacture process of the oxygen carriers, the metal oxides are combined with an inert which acts as a porous support providing a higher surface area for reaction, and as a binder for increasing the mechanical strength and attrition resistance [12-13]. Temperature variations in the oxygen carrier particles during their reduction and oxidation in a chemical-looping combustion system are equally important to the overall process. In this work, the inert solid used for the preparation of oxygen carriers have been considered Al_2O_3.

RESULTS AND DISCUSSION

This paper describes two chemical looping combustion systems of the carbon dioxide capture using a natural gas-based and a syngas-based chemical looping combustion system. The processes are identical in design; the difference is in terms of substance used as oxygen carrier and the fuel used (syngas and natural gas). The evaluated oxygen carriers are copper oxide (CuO, Cu_2O), nickel oxide (NiO) and manganese oxide (Mn_3O_4) [14-16]. The process is composed of two interconnected fluidized bed reactors as shown in Figure 1.

The fuel comes in contact with the oxygen carrier in the fuel reactor. The reduction of the oxygen carrier takes place at about 750-850°C, according to the chemical reactions (the first four reactions are for the case of natural gas used as fuel, the next reactions are for the case of syngas used as fuel):

$$4NiO_{(s)} + CH_{4(g)} \rightarrow 4Ni_{(s)} + CO_{2(g)} + 2H_2O_{(g)} \quad (3)$$
$$4CuO_{(s)} + CH_{4(g)} \rightarrow 4Cu_{(s)} + CO_{2(g)} + 2H_2O_{(g)} \quad (4)$$
$$4Cu_2O_{(s)} + CH_{4(g)} \rightarrow 8Cu_{(s)} + CO_{2(g)} + 2H_2O_{(g)} \quad (5)$$
$$4Mn_3O_{4(s)} + CH_{4(g)} \rightarrow 12MnO_{(s)} + CO_{2(g)} + 2H_2O_{(g)} \quad (6)$$
$$NiO_{(s)} + CO_{(g)} \rightarrow Ni_{(s)} + CO_{2(g)} \quad (7)$$
$$NiO_{(s)} + H_{2(g)} \rightarrow Ni_{(s)} + H_2O_{(g)} \quad (8)$$
$$CuO_{(s)} + CO_{(g)} \rightarrow Cu_{(s)} + CO_{2(g)} \quad (9)$$
$$CuO_{(s)} + H_{2(g)} \rightarrow Cu_{(s)} + H_2O_{(g)} \quad (10)$$
$$Cu_2O_{(s)} + CO_{(g)} \rightarrow 2Cu_{(s)} + CO_{2(g)} \quad (11)$$
$$Cu_2O_{(s)} + H_{2(g)} \rightarrow 2Cu_{(s)} + H_2O_{(g)} \quad (12)$$
$$Mn_3O_{4(s)} + CO_{(g)} \rightarrow 3MnO_{(s)} + CO_{2(g)} \quad (13)$$
$$Mn_3O_{4(s)} + H_{2(g)} \rightarrow 3MnO_{(s)} + H_2O_{(g)} \quad (14)$$

The stream which leaves the reactor is sent to a flash separator where the solid phase is separated from the gas phase. The rich CO_2 stream leaves the flash to the top and is sent to a heat recovery steam generation. The CO_2 hot stream is then cooled down with cooling water, at around 40°C, the temperature is achieved by using a series of heat exchangers (heat recovery steam generator - HRSG). The steam obtained in heat recovery steam generators by cooling the hot streams reaches a steam turbine where heat is converted into electricity. The exhausted steam leaves the turbine, then it is condensed and recycled back in the cycle (steam – Rankine cycle).

The CO_2 cooled stream is sent to a compression unit, which has 3 compression stages with intercoolers. In the first stage the gas is compressed from 14 bar to 20 bar. The stream is cooled at 40°C and enters in a flash separator, used to remove the water from the process. Cooling and separation are made after each stage of compression. In the second stage the pressure is increased from 20 bar to 70 bar, then the pressure reaches in the third stage 120 bar. The compression stages were used instead of one compressor because one compressor needs more energy to achieve 120 bar than 3 compression stages. Another argument is to avoid the overheating of the compressors. The solid stream coming from the bottom of the flash separator is sent to the air reactor where is oxidized with air at 850-950°C, according to the following reactions:

$$4Ni_{(s)} + 2O_{2(g)} \rightarrow 4NiO_{(s)} \qquad (15)$$
$$4Cu_{(s)} + 2O_{2(g)} \rightarrow 4CuO_{(s)} \qquad (16)$$
$$8Cu_{(s)} + 2O_{2(g)} \rightarrow 4Cu_2O_{(s)} \qquad (17)$$
$$3MnO_{(s)} + 1/2O_{2(g)} \rightarrow Mn_3O_{4(s)} \qquad (18)$$

The oxygen carrier obtained is recycled to the reduction reactor (fuel reactor). The gas stream leaves the cyclone to the top and is sent, like the CO_2 stream, to a heat recovery steam generator. The generated steam is then expanded in a steam turbine to produce the ancillary power. The natural gas and the syngas composition used in the mathematical modeling and simulation are presented in Table 1. ChemCAD was used as simulation software. As calculation model for simulation of the process, thermodynamic equilibrium was chosen, the thermodynamic package being selected considering the range of operating parameters (e.g. pressure, temperature) as well as the chemical species present in the system.

1. Syngas chemical looping combustion

The first process simulated used syngas as fuel. In order to evaluate the effect of the oxygen carrier flow on the reactor temperature Figure 2 shows the temperature curve in the fuel reactor due to the oxygen carrier flow. Various oxygen carriers were evaluated (CuO, Mn_3O_4, NiO and Cu_2O).

The results suggest a logarithmic influence between inlet oxygen carrier flow and the temperature, the temperature increase with the oxygen carrier (OC) load. The temperature tends to became stabile when the maximum conversion of the oxygen carrier is reached. This conclusion is important for the practical operation on the chemical looping unit.

Table 1. Fuel characteristics used in analysis

Fuel	Parameters	Values
Natural gas	Temperature (°C)	30.00
	Pressure (bar)	15.00
	Gas composition (% vol.)	
	Methane	89.00
	Nitrogen	0.89
	Carbon dioxide	2.00
	Ethane	7.00
	Propane	1.00
	I-Butane	0.05
	N-Butane	0.05
	I-Pentane	0.005
	N-Pentane	0.004
	Hexane	0.001
	Hydrogen Sulfide	0.00001
Syngas	Temperature (°C)	30.00
	Pressure (bar)	31.50
	Gas composition (% vol.)	
	Methane	0.01
	Nitrogen	5.14
	Carbon dioxide	4.47
	Carbon monoxide	61.34
	Hydrogen	28.09
	Water	0.02
	Argon	0.93

As shown in Figure 2, the temperature in the fuel reactor is influenced by the oxide used as oxygen carrier. With the increasing in amount of OC, from 10% OC to 90% OC (expressed in mass percentages from the total solid), the difference between the variation of the temperatures increase due to the reduced material flow through the reactor. This fact is important in order to find a suitable trade-off between the OC concentration and the temperature profile.

The Figure 3 shows the variation of temperature in the air reactor due to the air flow. The temperature in the air reactor decrease with the increasing of the air flow, because of the exothermic reaction in the reactor. As for the case of the fuel reactor, the solid material flow through the reactor influences the temperature profile.

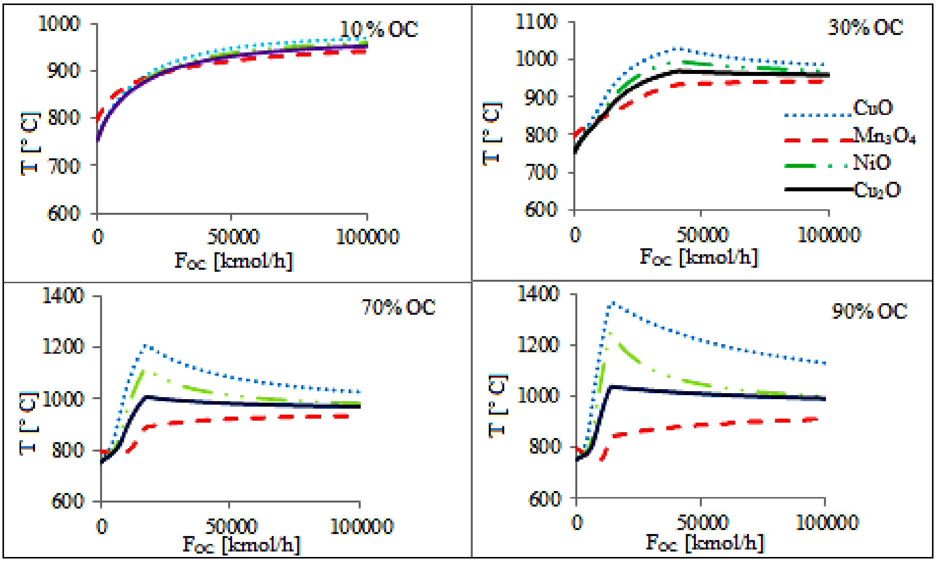

Figure 2. Variation of temperature in the fuel reactor due to the oxygen carrier flow with different concentrations

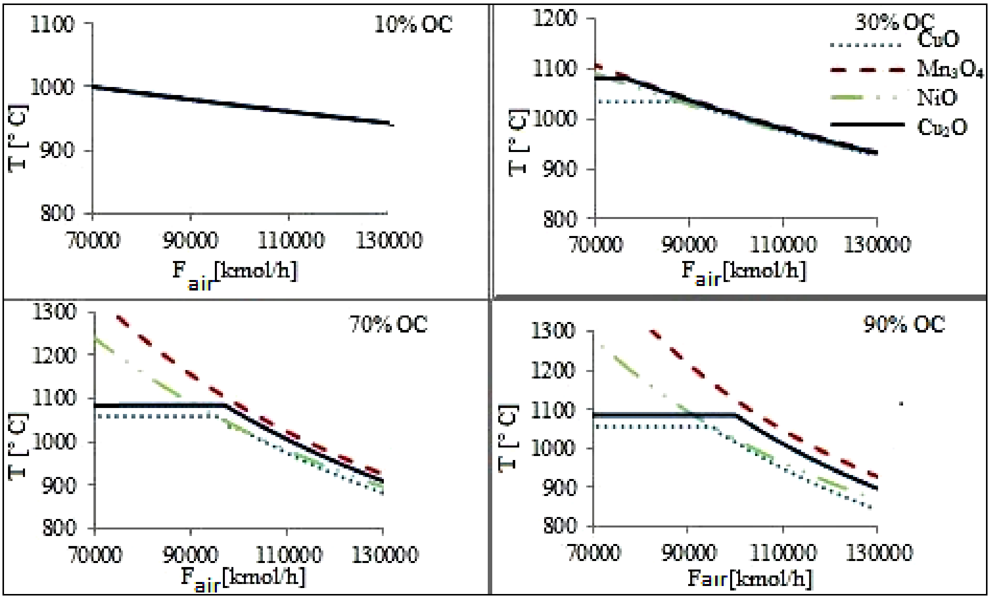

Figure 3. Variation of temperature in the air reactor due to the air inlet flow

In view of maintaining a sufficient fuel reactor temperature for a high conversion rate, CuO has been highly appraised and intensively studied for the exothermic characteristics of its reduction reaction with various fuels [17]. The exothermic character of both fuel and air reactors simplify the energy management of the whole process and increase the overall energy efficiency. In the following text, the evaluation was concentrated on using the copper oxide as oxygen carrier in chemical looping.

Figure 4 describes the variation of CO molar fraction due to oxygen carrier flow, at different pressures. The CO molar fraction decreases with the increasing of OC. The trends are similar with the increasing of the pressures, in the range of 2 to 32 bar. As can be noticed from Figure 4, the influence of pressure is rather limitate on the process.

A similar trend is observed also for hydrogen molar fraction (see Figure 5). As can be noticed from Figure 5, hydrogen molar fraction increases, reaches a maximum, and then the slope has a downward trend to zero (total conversion). Figure 5 shows also the influence of syngas pressure over the process, as the pressure increases the hydrogen oxidation process is more favorable. The influence of pressure for syngas looping combustion is of great importance considering that the most efficient gasification / catalytic reforming techologies are running at hight pressure (up to 40 bar).

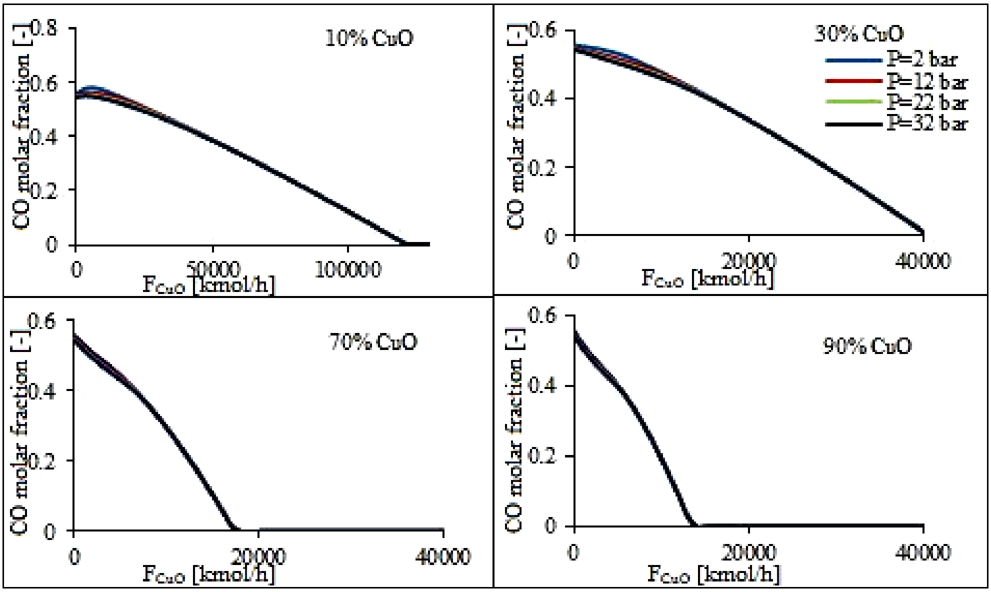

Figure 4. Variation of CO molar fraction as a function of CuO inlet flow

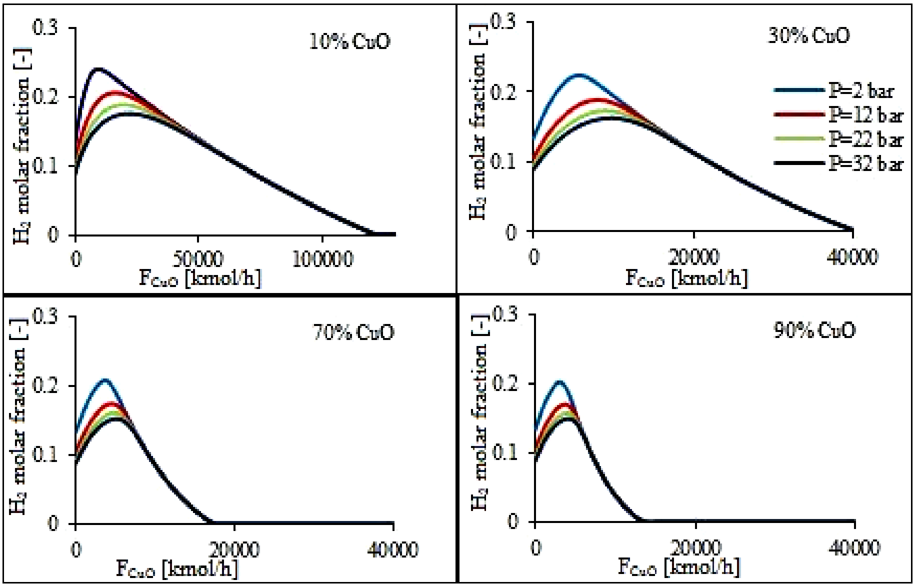

Figure 5. Variation of H_2 molar fraction as a function of CuO inlet flow

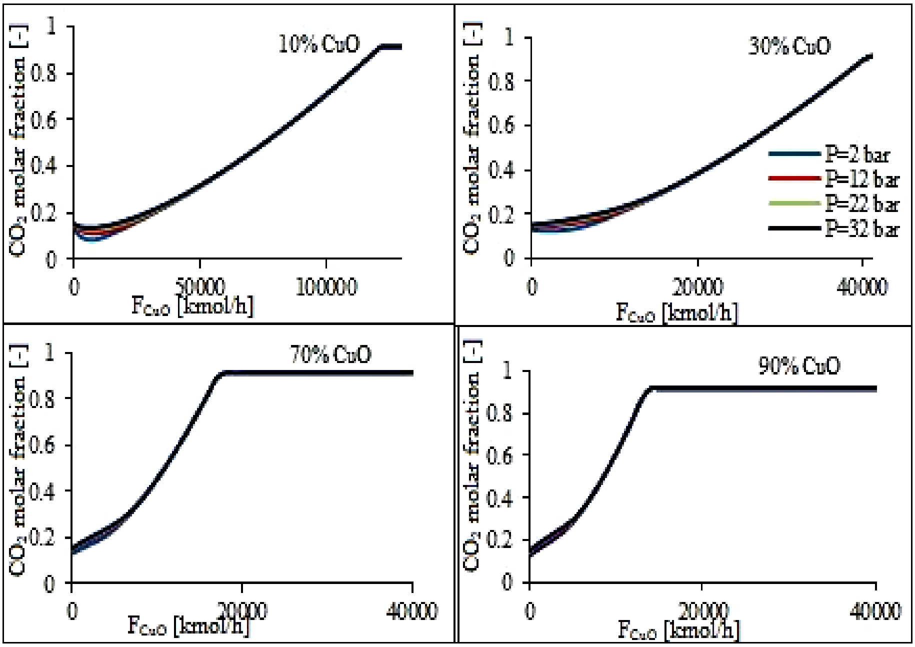

Figure 6. Variation of CO_2 molar fraction as a function of CuO inlet flow

The CO_2 molar fraction increases with the increasing of the CuO flow, the CO_2 fraction reaches 99%. The CO_2 is influenced by the pressure just at low flows of CuO. As presented in Figures 2 - 6, these numerical evaluations are of significant importance to establish the process conditions (pressure, temperature, solid and gaseous flows etc.).

2. Natural gas chemical looping combustion

Figure 7 illustrates the trend of the temperature in the fuel reactor, due to the oxygen carrier for natural gas-based chemical looping. The fuel reactor temperature increases with the increasing of OC. The temperatures tempt stability below 1000°C. The highest temperature in the fuel reactor is achieved using CuO as oxygen carrier, while the lowest temperature is obtained with Mn_3O_4 as OC. The temperature variation based on OC influence is visible with the decreasing of inert material in the OC inlet flow.

Figure 8 shows the variation of temperature in the air reactor due to the air flow. The temperature decreases with the increasing of the OC inlet flow. As presented already for syngas-based chemical looping combustion, the concentration of OC has an important influence on temperature profiles in both reactors. The operational conditions (e.g. oxygen carrier concentration in the solid phase) have to be modulate in a such a way to maximize the fuel conversion (which generally imply a temperature increase) but not to cause sintering of the solid and maintaining a suitable mechanical strenght to prevent particle fragmentation.

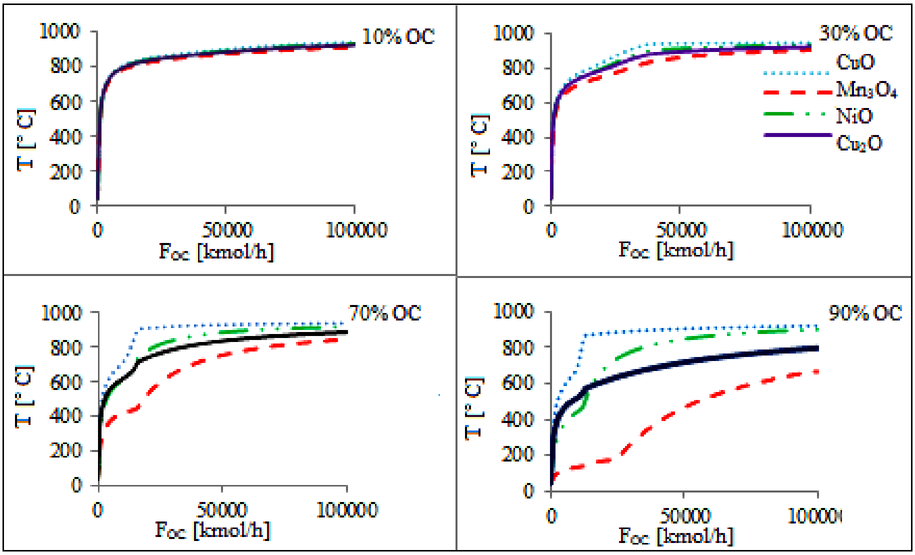

Figure 7. Variation of temperature in the fuel reactor due to the oxygen carrier flow

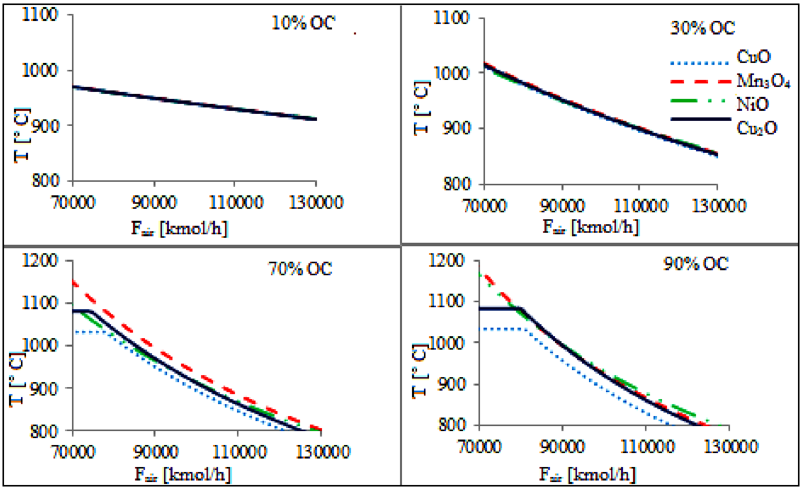

Figure 8. Variation of temperature in the air reactor due to the air flow

Figure 9 shows the variation of CH_4 molar fraction as a function of CuO inlet flow at different pressures. The CH_4 molar fraction decreases with the increasing in CuO. The influence of pressure was analysed in the range 2 to 32 bar. The oxygen carrier flow has to be selected in a such a way to ensure an almost total fuel oxidation. As can be noticed from Figure 9, the increase of pressure implies the increase of OC flow to ensure a total oxidation of the fuel.

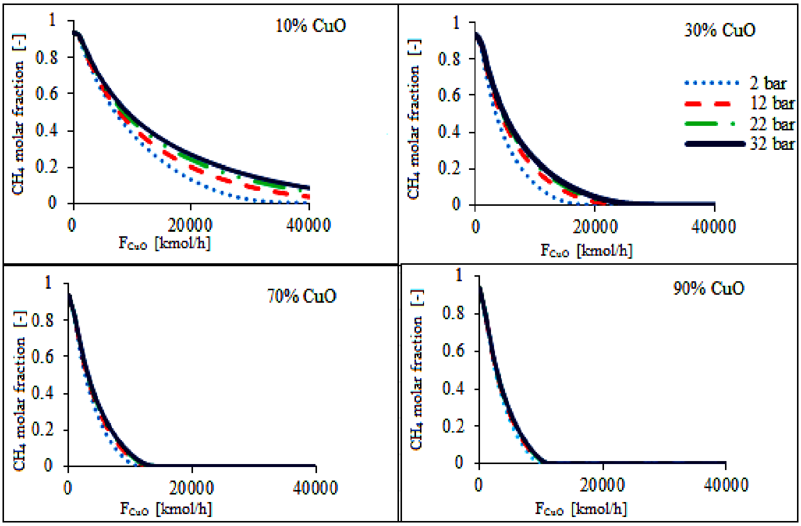

Figure 9. Variation of CH_4 molar fraction as a function of CuO inlet flow at different pressures

Figures 10-11 describe the variation of CO and H_2 molar fractions as a function of oxygen carrier (CuO) flow. These chemical species are formed in the natural gas oxidation process and then are they are finally oxidised to carbon dioxide and water. The behavior of carbon monoxide and hydrogen molar fractions is similar, the trend is to increase up to a maximum fraction, then they are reduced to a minimum concentration, close to zero. The molar fraction is influenced by the pressure, the increase of the pressure decreases the range in which these molar fractions varies. These results are important for evaluation of pressure influence on chemical looping combustion conditions.

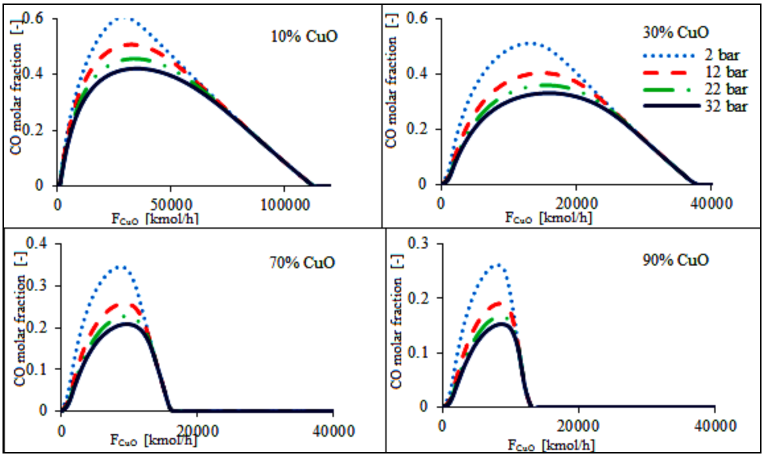

Figure 10. Variation of CO molar fraction as a function of CuO inlet flow

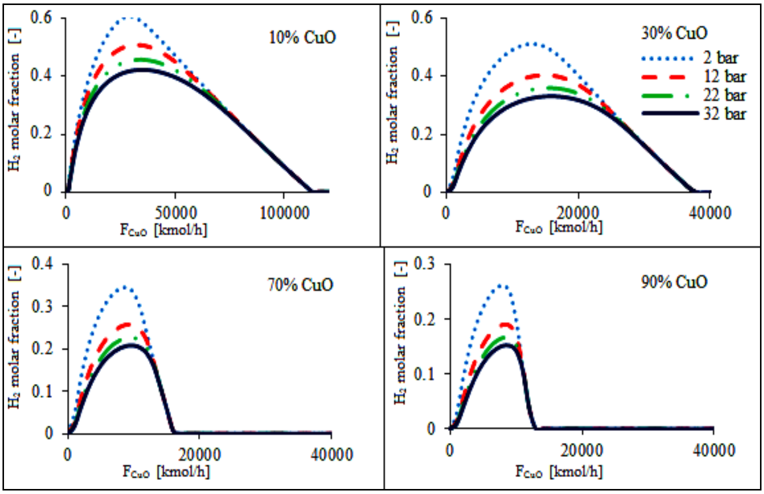

Figure 11. Variation of H_2 molar fraction as a function of CuO inlet flow

The Figure 12 describes the variation of CO_2 molar fraction as a function of CuO inlet flow at various pressures. The molar fraction increases with the increasing of the inlet flow of CuO. The trend is similar with increasing pressures, but without a significantly difference.

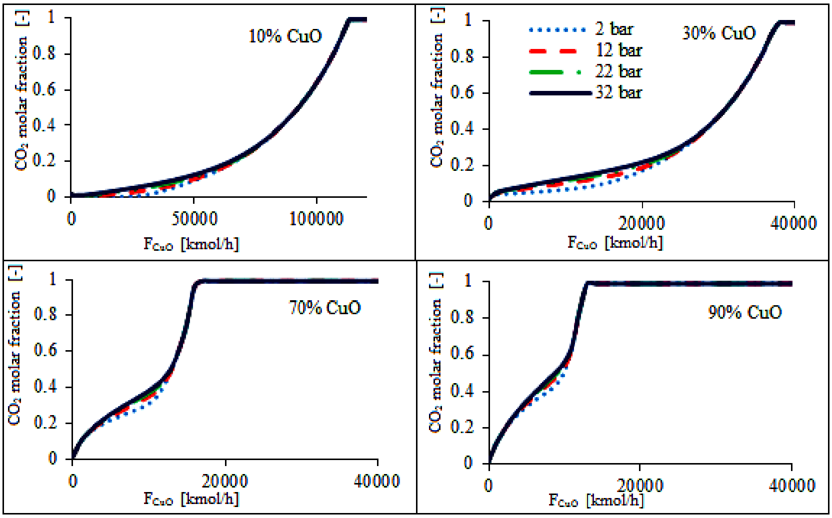

Figure 12. Variation of CO_2 molar fraction as a function of CuO inlet flow at different pressures

Table 2. Overall plant performance indicators

Main plant data	Units	Iron	Nickel
Natural gas flow rate	t/h	38.708	38.708
Natural gas LHV	MJ/kg	46.502	
Natural gas thermal input (A)	MW_{th}	500.00	500.00
Steam turbine	MW_e	164.13	164.21
Purge gas expander	MW_e	291.87	291.84
Gross power output (B)	MW_e	456.01	456.05
Ancillary power consumption (C)	MW_e	247.11	246.49
Net power output (D)	MW_e	208.90	209.56
Net electrical efficiency (D/A*100)	%	41.78	41.91
CO_2 capture rate	%	99.07	99.07
Specific CO_2 emissions	Kg/MWh	1.85	1.81

As presented in above evaluations, the numerical assessments (based mainly on thermodynamic factors) of various oxygen carriers for gaseous fuel chemical looping combustion are extremely useful for optimization of the experimental conditions to ensure both an almost total fuel conversion and a high energy efficiency. The optimised values of the process parameters can be then used for performance assessment of industrial-scale chemical looping combustion cases.

As an illustrative case, Table 2 presents the overall plant performance indicators for natural gas looping combustion using iron and nickel oxides as reported in a previous study [6], the evaluations being based on optimised process conditions as reported in this paper.

CONCLUSIONS

Chemical looping combustion is one promising techniques used to combine fuel combustion and pure CO_2 production in situ allowing for CO_2 sequestration. This technique has significantly higher energy efficiency and lower energy and cost penalties compared with other carbon capture methods (e.g. physical or chemical gas-liquid absorption in both pre- and post-combustion capture configurations).

This paper investigates the behavior of various oxygen carriers used for natural gas and syngas chemical looping combustion in terms of optimising process conditions e.g. temperature, pressure, molar ratio between the reactants, oxygen carrier composition etc. For instance, the ratio between the oxygen carrier and the support is of paramount importance for process conditions (e.g. reactor temperature and heat transfer between the fuel and air reactors) and overall performance indicators. The assessment was based on numerical methods using an thermodynamic approach for the calculations. The results of the evaluation can be successfully used for optimization of the process parameters (pressure, temperature, fuel to OC ratio, oxygen carrier concentration etc.) in order to maximize the fuel conversion and overall plant energy efficiency.

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