



GHGT-12

Design and operation of a coal-fired 50 kW_{th} chemical looping combustor

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Abstract

A 50 kW_{th} pilot plant for solid fuels have been designed and constructed to be operated in both *i*G-CLC and CLOU modes for solid fuel combustion. The design of the concept was based on two interconnected circulating fluidized beds reactors. A carbon stripper has been included between fuel and air reactors to increase the residence time of char particles in the fuel reactor, thus allowing high CO₂ capture rates. The char particles are separated and recirculated to the fuel reactor. In this device, the solid flow exiting from the fuel reactor splits into two different streams. One goes to the carbon stripper and the other is recirculated to the fuel reactor. In this way it is possible to have an independent control of solid inventory in the fuel reactor and the global solid circulation flowrate between fuel and air reactors. Startup and experimental results obtained in this unit during combustion of a subbituminous coal with ilmenite by *i*G-CLC are presented. Low oxygen demand values were found (5.2%) in gases coming from the fuel reactor. This value agrees with the predictions obtained with a previously validated model. Moreover carbon capture efficiencies of 95% were found due to the high separation efficiency of the carbon stripper.

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1. Introduction

In Chemical Looping Combustion (CLC) with solid fuels, the fuel is physically mixed with the oxygen carrier. A scheme of the CLC with solid fuels is shown in Fig. 1. The oxygen needed for fuel combustion is supplied by an oxygen carrier, normally a metal oxide, which circulates between the so-called fuel and air reactors. In the fuel reactor, the fuel is oxidized to CO_2 and H_2O , which facilitates CO_2 capture once the water has been condensed. The reduced oxygen carrier is then transported to the air reactor where it is re-oxidized before starting a new cycle. The net chemical reaction and combustion enthalpy is the same as in a conventional combustion. CO_2 capture is inherent to this process, as the air is not mixed with the fuel. Significant advances have been made in the development of CLC using solid fuels in recent years by means of two processes: in-situ gasification CLC (*iG-CLC*) and Chemical Looping with Oxygen Uncoupling (CLOU) [1].

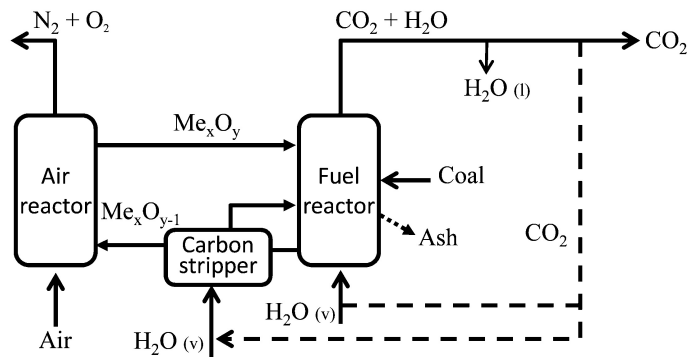


Fig. 1. Reactor scheme of CLC process with solid fuels.

In the *iG-CLC* concept coal is fed directly to the fuel reactor and mixed with the oxygen carrier. Steam and/or recycled CO_2 are supplied to the fuel reactor as fluidizing agents. The in-situ gasification of coal happens, generating volatile matter and gasification products, which are oxidized by gas-solid reactions with the oxygen carrier. Gasification is the limiting step for coal conversion. The slow coal gasification rate may cause that the solids stream exiting from the fuel reactor contained some unconverted char together with the oxygen carrier and ash. Once the unconverted char reaches the air reactor it reacts with air to produce CO_2 , which could decrease significantly the CO_2 capture efficiency of the process. To increase the residence time of char particles in the fuel reactor, without excessively increasing the reactor size, a carbon separation unit after the fuel reactor is included. In this unit, char particles are separated from the oxygen carrier particles and recirculated to the fuel reactor using their different fluidization properties. However, complete combustion of gases from the fuel reactor was not reached in existing *iG-CLC* units. Some strategies have been proposed to convert the unburnt compounds. They include an oxygen polishing step to complete gas combustion, the separation and recirculation of unburnt compounds or a secondary fuel reactor in series [2].

The CLOU concept was developed based on the use of oxygen carrier materials which can release gaseous oxygen and thereby allowing the solid fuel to be burnt with gas phase oxygen [3]. These materials can be also regenerated at high temperatures. Complete combustion and CO_2 capture rates close to 100% has been found in a 1.5 kW_{th} CLOU unit due to the fast combustion of coal with gaseous oxygen generated in-situ in the fuel reactor [4].

To date, several design concepts have been developed for *iG-CLC* ranging from 10 kW_{th} to 3 MW_{th} units, but no operation at scale higher than 1.5 kW_{th} is found for CLOU [1]. Major differences between units were found in the fuel reactor design. Smaller units include bubbling- or spout-fluid beds as fuel reactors, whereas bigger units are designed to operate at the high velocity fluidized bed regime. Thus, the cross-sectional area per power unit is minimized. Markström et al. [5], Orth et al. [6], and Andrus et al. [7] adopted the concept consisting on two interconnected circulating fluidized beds in *iG-CLC* pilots of 100 kW, 1 MW and 3 MW, respectively.

The 100 kW_{th} unit [5] allowed stable operation with ilmenite and CO₂ capture rate values close to 100% were obtained because of the high efficient carbon stripper included [8]. It is characterized by a low solid inventory in the fuel reactor (300–400 kg/MW_{th}), which results in a relevant fraction of unconverted gases in the fuel reactor. Note that theoretical calculations recommended values ranging from 750 to 1500 kg/MW_{th} in order to have a trade-off between combustion efficiency and ilmenite inventory [9,10]. The solids inventory in the fuel reactor and the solids circulation rate is uncoupled by using a circulation riser. However, a relatively high steam flow is required in the carbon stripper and the circulation riser, which result in high values, between 3 and 4, of steam to fixed carbon ratio.

The 1 MW_{th} unit [6] was designed for solids inventory values about 200–300 kg/MW_{th}. The solids circulation flow rate is modified by controlling the fraction of solids entrained from the air reactor which are sent to the fuel reactor. Thus, the amount of solids in the fuel reactor would be directly linked to the solids circulation rate. During preliminary operation, steam to fixed carbon ratio was about 2.

The 3 MW_{th} unit [7] was characterized by a direct hydraulic link between the bottom of fuel and air reactors. The hydraulic link allowed reaching high CO₂ capture rates without the necessity of a carbon stripper. This system is similar to that found in a 120 kW_{th} CLC unit for gaseous fuels [11], where the amount of solids in the fuel and air reactors were closely dependent on the gas velocity and the solids circulation flow rate.

The objective of this work was to design and built a flexible CLC 50 kW_{th} unit for solid fuels, which could be operated both in *i*G-CLC and CLOU modes. The design of the concept is based on two interconnected circulating fluidized beds reactors. Independent control of solids circulation flow rate and solids inventory in the fuel and air reactors is intended with the proposed design. Thus, direct relation between operational conditions and performance of the CLC unit could be easily evaluated.

A carbon stripper to improve carbon capture efficiency by recovering char particles that had escaped from the fuel reactor is included. The design includes a double loop seal after the fuel reactor cyclone to separate the reduced oxygen carrier and char into two different streams. One stream goes to the carbon stripper and the other one is recycled to the riser of the fuel reactor. In this way it is possible to reach one independent control of solid circulation in fuel reactor from the global solid circulation flow. Moreover, the measurement of solids circulation between fuel and air reactors can be performed by means of two diverting solid devices located down the cyclones. This unit allows investigating the performance of the *i*G-CLC and CLOU processes burning different fuels (lignite, bituminous coal and biomass) using different oxygen carrier materials (Fe-based minerals or copper-based oxygen carrier). Preliminary results obtained during combustion of a subbituminous coal with ilmenite by *i*G-CLC are presented showing carbon capture efficiencies and oxygen demands.

2. Design of the 50 kW_{th} CLC unit with solid fuels

The design of the CLC unit for solids fuels is shown in Fig. 2. It is based on two interconnected circulating fluidized bed reactors, the air and fuel reactor, and a carbon stripper, being a bubbling fluidized bed. Ilmenite for *i*G-CLC ($d_p=170\ \mu\text{m}$; $\rho_p=3710\ \text{kg/m}^3$) and a Cu-based material (60 wt.% CuO and 40 wt.% MgAl₂O₄; $d_p=170\ \mu\text{m}$; $\rho_p=3860\ \text{kg/m}^3$) for CLOU are used as oxygen carriers for design calculations. The solids inventory in the fuel reactor was set to 20 kg in *i*G-CLC, corresponding to 1000 kg/MW_{th}. This solids inventory value was estimated as the amount of oxygen carrier to balance the trade-off between the increase of pressure drop and the decrease in oxygen demand caused by an increase of solids in the reactor [10]. In CLOU, the same amount of solids corresponded to 400 kg/MW_{th}, which has been considered a suitable value from previous work [4]. For that design conditions the nominal thermal power was 20 kW_{th} for *i*G-CLC mode and 50 kW_{th} for CLOU mode. The difference of nominal power is because the better performance of CLOU with respect to *i*G-CLC on the basis of CO₂ capture and combustion efficiency [12].

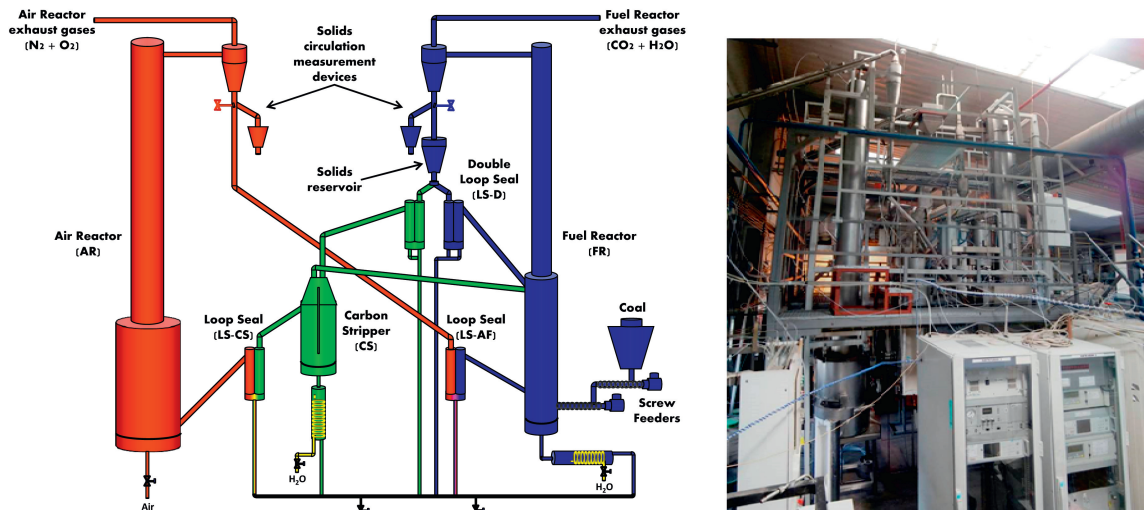


Fig. 2. Layout and a picture of the 50kW_{th} CLC Pilot Plant facility at ICB-CSIC-s50.

Different types of coals, ranging from lignite to anthracite, as well as biomass could be used as fuel. Mass balances, fluid dynamics considerations and the performance of the fuel reactor predicted by a theoretical model previously developed [9] were taken into account for the design of the CLC unit. Thus, solids circulation flow rate, reactors temperature, solids inventory in the fuel reactor, steam requirements and air flow were determined. Also general considerations about the carbon stripper, loop seals, and the cyclone system are discussed.

The design was done on the basis of the operation both in *i*G-CLC and CLOU modes. The solid fuel feeding point was just above the distributor plate in the fuel reactor. It is intended to convert the fuel to CO₂ and H₂O, minimizing the unburnt compounds. To maximise the solid fuel conversion, the fuel reactor temperature was considered to be 1000°C in *i*G-CLC [13] and 950°C in CLOU [4]. The CLC unit is not expected to be auto-thermal due to heat losses associated to the unit size. Then, the fuel reactor, air reactor, carbon stripper, and loop seals are heated by separate furnaces.

The dimensions of the reactor are defined by the design values of the pressure drop, solids inventory and gas velocity. Design data are shown in Table 1. The cross sectional area corresponded to 0.39 m²/MW_{th} in *i*G-CLC and 0.16 m²/MW_{th} in CLOU. The fuel reactor can be fluidized by H₂O, CO₂ or mixtures of them. The inlet gas velocity was just above the terminal velocity of oxygen carrier particles; see Fig. 3. When the gases from coal conversion together gas stream from the carbon stripper were considered, the gas velocity was estimated to be 4 m/s in *i*G-CLC and 5.5 m/s in CLOU. Under these conditions, the solids flux to the fuel reactor cyclone was estimated to be 30 kg m⁻² s⁻¹ in *i*G-CLC and 50 kg m⁻² s⁻¹ in CLOU [11], which are higher values than those required for stoichiometric conditions, i.e. 7.5 kg m⁻² s⁻¹ in *i*G-CLC and 12.5 kg m⁻² s⁻¹ in CLOU. Stoichiometric conditions were defined by a value of the oxygen carrier to fuel ratio $\phi = 1$. The ϕ parameter was defined as the flow of oxygen available in the circulating solids stream divided by the flow of oxygen required to achieve complete fuel combustion.

The maximum oxygen carrier to fuel ratio achievable in the CLC unit was estimated to be $\phi = 4$. However, ϕ values between 1 and 2 are preferable to maximize the CO₂ capture rate. For design conditions ϕ was selected to be 2, which means that the solids circulation flow to the air reactor must be lower than the solids flow exiting the fuel reactor. Thus, the double loop seal (LS-D) must be operated to fit the solids circulation towards the carbon stripper, and subsequently to the air reactor, at the desired value. The LS-D system is designed in order to divide the solids stream from the fuel reactor cyclone in two: (1) solids to the carbon stripper, and then to the air reactor, in order to fulfill the required solids circulation flow rate between air and fuel reactors; and (2) excess of solids was recirculated to the fuel reactor. In this way, the residence time of solids, including unconverted char particles, is maximized whereas the solids circulation flow rate between the air and fuel reactors was guaranteed. The use of a double loop

seal was considered to be an improvement with respect to existing CLC units for solid fuels. Another important point is the possibility to have a direct measurement of the solids flux exiting from both the fuel and air reactor by means of the respective solid circulation measurement devices.

Table 1. Main design parameters of the 50kW_{th} Pilot Plant.

	iG-CLC			CLOU		
	FR	AR	CS	FR	AR	CS
Power (kW)	20			50		
Height (m)	4.15	4.80	0.71	4.15	4.80	0.71
Diameter (cm)	10.2/8.1	30.0/10.2	15.0	10.2/8.1	30.0/10.2	15.0
Solids inventory (kg)	20	55	8	20	55	8
ΔP (kPa)	25	15	4.5	25	15	4.5
u _{g, in} (m/s)	0.50	0.40	0.35	0.50	0.90	0.35
u _{g, out} (m/s)	4.00	4.00	0.35	5.50	9.00	0.35
Stoichiometric G _{S,FR} (kg·m ⁻² ·s ⁻¹)	7.5	-	-	12.5	-	-
Designed G _{S,FR} (kg·m ⁻² ·s ⁻¹)	30	11	-	50	18	-

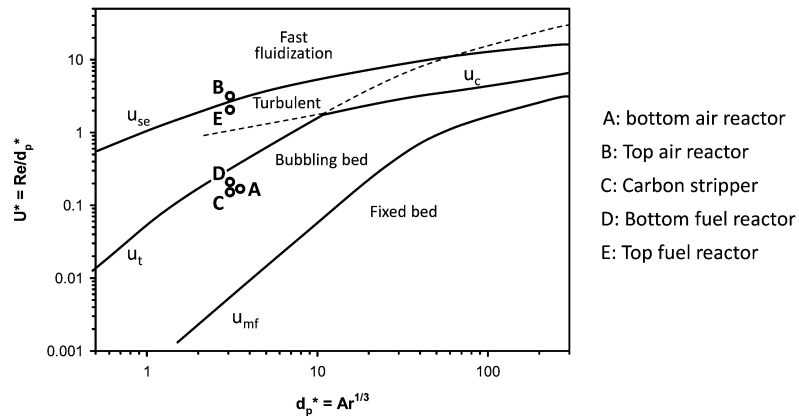


Fig. 3. Fluidization regime in the flow regime map [14].

The carbon stripper separates the unconverted char particles from the oxygen carrier according to their different fluid dynamic properties. Char particles are lighter and smaller than oxygen carrier particles. So, char particles can be elutriated from the carbon stripper and recirculated to the fuel reactor using an appropriate fluidization flow, whereas the oxygen carrier particles are left to pass to the air reactor via loop seal LS-CS. Char particles are recirculated to the bottom bed of the fuel reactor, but well above the distributor plate. It is preferable the use of CO₂ to fluidize this reactor, thus minimizing the steam duty of the CLC unit. Temperature in carbon stripper was set to 950°C. There are not yet works optimizing the design of a carbon stripper to minimize the solids inventory, whereas high carbon separation efficiency was reached. Here, the carbon stripper was designed as a bubbling bed 0.3 m height and 0.15 m diameter, and residence time of solids was estimated to be 100 s when φ = 2. The gauge pressure at the carbon stripper must also account the pressure drop in the riser of the fuel reactor. Considering the inlet point of solids entrained from the carbon stripper to the fuel reactor, the additional pressure drop to be overcome in the fuel reactor was 10 kPa. The gas velocity in this unit must be higher than terminal velocity of char particles (u_{t,char} = 0.1 m/s), but lower than terminal velocity of oxygen carrier particles (u_{t,OC} = 1 m/s); see Fig. 3. A gas velocity in the carbon stripper of 0.35 m/s seems to be suitable to reach an effective separation of char particles from oxygen carrier particles.

In the air reactor, the oxygen carrier was regenerated with air. The air reactor was designed to achieve complete oxidation of the oxygen carrier (residence time $t_{mr} = 700$ s), whereas the solids circulation flow rate must be guaranteed. Thus, a wide bottom bed was followed by a narrow riser. The air flow was set with a 10% excess with respect to that needed to burn the fuel fed in the fuel reactor. A low gas velocity was chosen in the bottom bed (0.4 m/s in *i*G-CLC) to operate in the bubbling fluidization regime. The gas velocity increased up to 4 m/s in the riser because the lower diameter, which allowed to operate in the fast fluidization regimen; see Fig. 3. The solids flux in the riser should be $11 \text{ kg m}^{-2}\text{s}^{-1}$ for $\phi = 2$, which may be easily reached under these conditions. This means that the solid flux in the air reactor is determined by the solids flux entering from the carbon stripper, which depends on the double loop seal operation. Some higher velocities were required in CLOU, 0.9 m/s in the bottom bed and 9 m/s in the riser, because the unit was designed for a higher thermal power in this mode.

Solids entrained from air and fuel reactors were separated by respective cyclones and sent to the next element in the CLC unit. The dimensioning of the cyclone was done on the basis of a high efficiency standard cyclone.

Loop seals avoid the mixing of gases between different elements in the CLC unit. Thus, loop seal LS-CS avoids the mixing of air and $\text{H}_2\text{O-CO}_2$ in the carbon stripper, whereas loop seal LS-AF hinders the mixing of air and gases from fuel. Loop seal LS-D is necessary to avoid the malfunction of cyclone in the fuel reactor because leaking of gas from the carbon stripper to this cyclone. Moreover, this loop seal controls the circulation flow rate of solids between the fuel and the air reactors. The loop seal units were designed as bubbling fluidized beds and were dimensioned to allow a high solids circulation flow rate, i.e. the loop seal must not limit the solids circulation flow rate. Thus, the required solid flux in loop seals would be $16 \text{ kg m}^{-2}\text{s}^{-1}$ at the oxygen carrier to fuel ratio $\phi = 2$, whereas the maximum capacity could be as high as $300 \text{ kg m}^{-2}\text{s}^{-1}$. Loop seals must also balance the pressure between the elements it connects. Thus, the length of the low pressure hand side of each loop seal is given by the pressure balance in the system, as it is shown in Fig. 4.

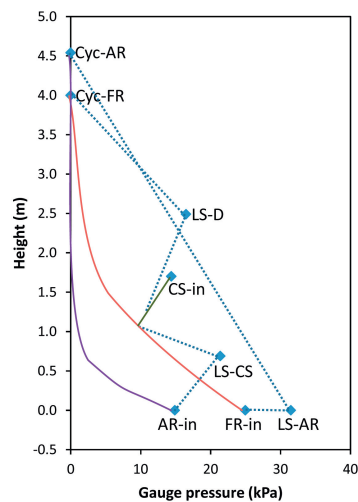


Fig. 4. Estimated pressure profile in the CLC unit. Dotted lines are elements connected for different loop seals. Continuous lines: pressure evolution with height in each element of the CLC unit.

The LS-D must balance an overpressure of 10 kPa in the fuel reactor inlet point from the carbon stripper with respect the fuel reactor cyclone exit. LS-CS must balance a low difference on pressure between the bottom of the air reactor and the upper part of the carbon stripper. LS-AF must be carefully designed to balance a high pressure difference between the air reactor cyclone and the bottom part of the fuel reactor, which is 25 kPa. The difference of pressure between both sides of each loop seal must be compensated by a solids column in the side with lower pressure, which must be at least 0.8, 0.4 and 2.0 m for loop seals LS-D, LS-CS and LS-AF, respectively. Estimated pressure drops in each element of the CLC unit for the design conditions can be calculated following continuous lines in Fig. 4. Note that the pressure at the reactors inlet should account for an additional pressure drop of 30% due

to the presence of a distributor plate in the fluidized beds. Finally, the height of air and fuel reactors was determined in order to arrange all the elements of the CLC unit, including the required column of solids in loop seals. Thus, the air reactor height was 4.65 m, whereas the fuel reactor was 4.0 m tall.

The performance of the CLC unit with coal as fuel was simulated by using theoretical models for *i*G-CLC and CLOU previously developed and validated against experimental results in 100 kW_{th} *i*G-CLC and 1 kW_{th} CLOU units [8,15]. “El Cerrejón” bituminous Colombian coal was assumed to be the fuel, which was used in previous experimental works [12]. Fig. 5 shows the CO₂ capture efficiency and the oxygen demand predicted in the CLC unit as a function of the carbon stripper efficiency separating unconverted char particles from the oxygen carrier stream. The oxygen demand was defined as the ratio of stoichiometric oxygen flow required to fully oxidize unconverted gases from the fuel reactor to the stoichiometric oxygen flow for complete coal combustion.

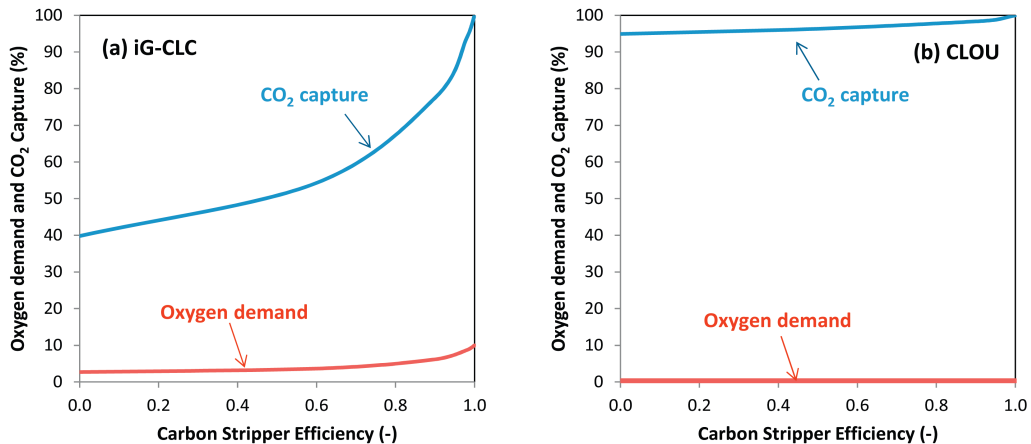


Fig. 5. Estimated CO₂ capture and oxygen demand in the CLC unit for (a) 20 kW *i*G-CLC; and (b) 50 kW CLOU.

A highly efficient carbon stripper is a key element in order to reach high CO₂ capture rates in *i*G-CLC mode, because of the slow coal gasification. Moreover, complete combustion was not predicted in the fuel reactor. Obviously, the oxygen demand increased as the fuel conversion in the fuel reactor increased. Thus, the oxygen demand in *i*G-CLC increased from 3% to 10% when the carbon stripper efficiency increased from 0 to 1. The estimated CLOU performance was much better than that predicted for *i*G-CLC. Thus, complete combustion was predicted even when the fuel power was higher (50kW_{th}) compared to those value assumed for *i*G-CLC (20 kW_{th}). Moreover, the fast coal conversion in CLOU via combustion with gaseous oxygen evolved from the oxygen carrier makes that CO₂ capture rates higher than 95% could be reached even if a poor carbon stripper performance was assumed. These differences between *i*G-CLC and CLOU agree to results previously described from experimental works in a 1 kW CLC unit [12].

3. Pilot operation

Experimental campaign in this CLC unit is ongoing using ilmenite as oxygen carrier and a South African bituminous coal. Table 2 shows main characteristics of ilmenite and coal used. Table 3 shows the main operating conditions used in the CLC unit. Temperatures of fuel and air reactor were 950 and 960°C; fuel reactor and carbon stripper were fluidized with steam, although N₂ was used initially used in the loop-seals.

Fig. 6 shows the evolution of temperatures and gas concentrations in fuel and air reactors during start-up and operation until steady state is reached for these operating conditions. During initial electrical heating all reactors were fluidized with air. After temperatures in fuel and air reactors were reached the fluidizing gas was changed to steam in the fuel reactor and in the carbon stripper. Then started the coal feeding into the fuel reactor and started the combustion by *i*G-CLC. However, the amount of oxygen reacted in the air reactor was low due the low temperature

existing in this reactor and ilmenite was not regenerated. This drives to one gradual decrease of the oxygen available for coal combustion in the fuel reactor. By this reason operating conditions in the carbon stripper were modified in order to minimize char separation to send char to the air reactor. In this way temperature of the air reactor was increased until fixed conditions. After carbon stripper was normally operated steady state was reached.

Table 2. Main characteristics of coal and ilmenite.

SA bituminous coal	
Moisture	3.5
Volatile matter	25.5
Ash	15.7
Fixed carbon	55.3
C (%)	66.3
H (%)	3.6
N (%)	1.8
S (%)	0.5
LHV (kJ/kg)	24930
d_p (μm)	+100-300
Ilmenite	
R_0 (%)	4.0
d_p (mm)	+100-300

Table 3. Operating conditions in *i*G-CLC.

Fuel Reactor	
Inventory of oxygen carrier (kg/MW _{th})	1100
Coal feed rate (kg/h)	1.7
Oxygen carrier to fuel ratio (Φ)	2
T (°C)	960
Fluidizing gas	Steam
u_{in} (m/s)	0.6
u_{out} (m/s)	3.8
Air reactor	
T (°C)	950
u_{in} (m/s)	0.6
u_{out} (m/s)	3.5
Fluidizing gas	Air
Carbon stripper	
Fluidizing gas	steam
U_0 (m/s)	0.4

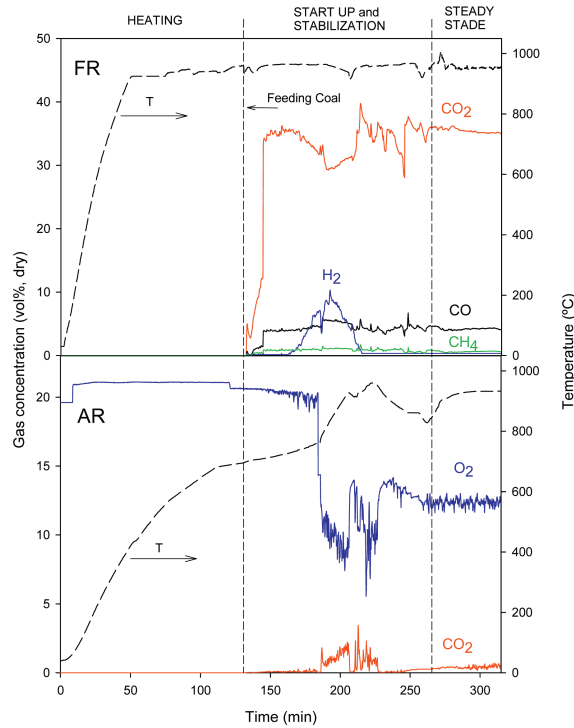


Fig. 6. Evolution of temperature and gas concentrations in the fuel and air reactors during pilot operation by *iG-CLC*.

Fig. 7 shows the evolution with time of the CO₂ capture and the total oxygen demand in gases coming from the fuel reactor. A value of 94.7% was found for the carbon capture efficiency. Using the model predictions the carbon separation efficiency of the carbon stripper can be determined using Fig. 5(a). Although the operation of the carbon stripper was not yet optimized, a value of 98% was found for this parameter. Total oxygen demand found was low, 5.6%, and it is in agreement with model predictions. This low value is due to the use of one inventory in the fuel reactor of 1100kg/MW_{th} which was established in a previous investigation to minimize oxygen demand [8].

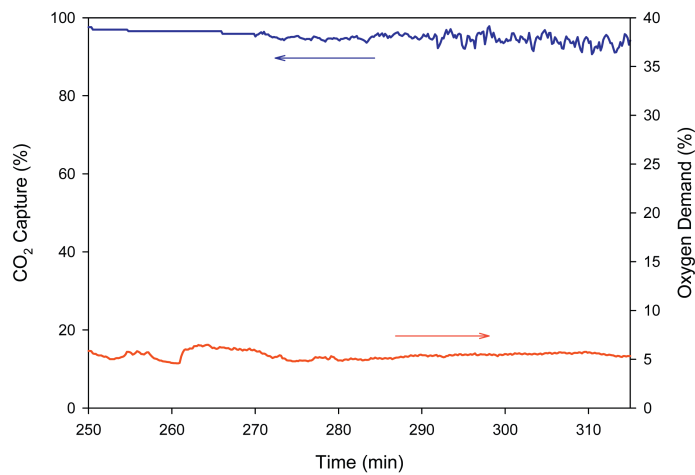


Fig. 7. CO₂ capture and oxygen demand in the CLC unit for burning one bituminous coal with ilmenite.

4. Conclusions

A CLC unit for solid fuels was designed to be operated in both *i*G-CLC and CLOU. The unit includes a fuel reactor, an air reactor and a carbon stripper. The use of a double loop seal down the fuel reactor cyclone must allow controlling the solids circulation flow rate independently of the solids inventory in the fuel reactor. Initial experiments carried out by *i*G-CLC burning a bituminous coal with ilmenite showed high CO₂ capture efficiencies with low oxygen demands for the gases coming from fuel reactor, in accordance with a model developed for this system.

Acknowledgements

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