

1 MW_{th} LONG-TERM PILOT TESTING OF THE CARBONATE LOOPING PROCESS WITH HARD COAL AND LIGNITE

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Abstract – The calcium carbonate looping (CaL) process is an efficient post-combustion carbon capture technology to reduce the emitted amount of carbon dioxide (CO₂) by fossil fueled power and industrial plants. The presented work discusses the results gained in four test campaigns, each four weeks long, performed in a 1 MW_{th} CaL pilot plant at Technische Universität Darmstadt with coal originated flue gas from a coal fired furnace. The calciner was fired in oxy-fuel combustion mode, i.e. coal was burned with oxygen and flue gas was recirculated to moderate the temperature. The objective of these test campaigns was to improve the process and to gain reliable experimental data to scale up the CaL process to industrial size. The type of fuel, sorbent, flue gas composition, reactor design, and operating conditions were varied to investigate long-term effects on the performance during pilot operation. Steady-state conditions were achieved while parameters were not changed during periods up to 60 hours. The feasibility of the CaL process in industrial size was proven by steady-state CO₂ absorption for more than 1,219 hours with interconnected circulating fluidized bed reactors and capture rates up to 94 %.

Keywords – calcium looping, CO₂ capture, pilot plant, dual fluidized bed, oxy-fuel regenerator

INTRODUCTION

An increase of the energy demand for the next decades is expected and fossil fuels as coal and natural gas will remain a major energy source for power production to cover the need (OECD/IEA, 2015a). The availability in large quantities in many areas worldwide ensures the power supply. Nevertheless, the knowledge about climate changes increased during the previous decades and it has been proven that global warming is mainly caused by the anthropogenic release of greenhouse gases. The effect of the greenhouse gas CO₂, inevitably formed and released in significant amounts by combusting fossil fuels, and its contribution to the global warming is generally accepted (OECD/IEA, 2015b). Carbon Capture and Storage (CCS) has been identified as an important contribution for mitigation of CO₂ emission of fossil fueled power plants (European Commission, 2015; OECD/IEA, 2015a). Various CO₂ capture processes to reduce the amount of CO₂ emitted by fossil fueled power and industrial plants have been proposed, developed and investigated. First generation CO₂ capture technologies, e.g. Mono-Ethylene-Amine Scrubbing (MEA, post-combustion), Integrated Gasification Combined Cycle (IGCC, pre-combustion) or Oxy-Fuel Combustion have the disadvantage of crucial net efficiency losses in a range of 9-14 %-points (Abu Zahra et al., 2011; Martelli et al., 2009). Hence, various second generation CO₂ capture processes are currently being developed aiming at improved efficiencies. The CaL process is characterized by a low net efficiency penalty of 5-7 %-points including CO₂ compression, low CO₂ capture costs below 30 €/t_{CO2} and low environmental impact (Epple and Ströhle, 2008). The CaL process has also aroused the interest of the cement industry during the last decade since spent sorbent based on the limestone used in the CO₂ capture process could be utilized as raw material for clinker production.

The CaL process is a post-combustion CO₂ capture technology using limestone based sorbents and was initially proposed by Shimizu et al. (1999). The CO₂ contained in the flue gas from an emission source is absorbed by calcium oxide (CaO) in the carbonator in an exothermic reaction at around 650 °C and transferred as calcium carbonate (CaCO₃) to the calciner. By increasing the temperature up to around 900 °C, the CO₂ bound in the solid phase, is released in an endothermic reaction. The heat for the endothermic calcination reaction can be provided in the directly heated CaL process by oxy-fuel combustion of fuel (e.g. coal) or in the indirectly heated CaL process (IHCaL) via heat transfer (e.g. heat pipes) from another reactor (Reitz et al., 2016). A gas stream of highly concentrated CO₂ leaves the calciner, while the CaO is returned to the carbonator. The reaction can be described by Eq. (1):



Since the basis concept was proposed, various research groups worldwide spent effort in the investigation of the CaL process and various pilot plants with increasing capacity were built. At the University of Stuttgart, a

200 kW_{th} pilot plant was raised. The Institute for Energy Systems and Technology (EST) at Technische Universität Darmstadt erected a 1 MW_{th} CaL pilot plant. The first continuous 1 MW_{th} CaL pilot scale operation in an interconnected circulating fluidized bed (CFB) system was achieved. Additional test campaigns were conducted using natural gas or coal in the calciner (Ströhle et al., 2014). Also a 1.7 MW_{th} CaL pilot plant was built in La Pereda (Spain) decarbonizing flue gas from a nearby power plant achieving high capture rates (Arias et al., 2013).

The present paper summarizes the main experimental results from four long-term CaL test campaigns carried out in the 1 MW_{th} pilot plant at Technische Universität Darmstadt with the aim to optimize operability as well as to gain a reliable data base for process scale-up to industrial size. The experimental tests were particularly focused on the long-term sorbent reactivity in realistic operating conditions such as oxy-fuel combustion in the calciner to guarantee a high CO₂ concentration in order to gain reliable information about the sorbent deactivation during various capture cycles of the limestone at realistic calcination conditions. During the test campaigns, the type of fuel (hard coal, lignite in various particle size distributions), the sorbent, flue gas composition, reactor design and operating conditions, e.g. reactor temperatures, make-up flow, solids circulation flow etc., were varied to investigate long-term effects on the performance (Helbig et al., 2016). Steady-state operation decarbonizing the flue gas of a 1 MW_{th} coal fired furnace was investigated during 2,400 hours of operation, thereof 1,219 hours with continuous CaL operation capturing CO₂.

EXPERIMENTAL

A semi-industrial scale CaL pilot plant consisting of two interconnected circulating fluidized bed reactors and a combustion chamber with a thermal capacity of 1 MW_{th} each is located at Technische Universität Darmstadt. The scheme of upgraded 1 MW_{th} CaL pilot plant at Technische Universität Darmstadt is shown in Fig. 1. The carbonator has an inner diameter of 600 mm and is 8.6 m high. The dimensions of the calciner are 400 mm and 11 m, respectively. The plant design is illustrated in Fig. 1. The plant is equipped with all conventional components of typical industrial CFB systems. The combustion chamber provides the coal originated flue gas for the carbonator. The mass flow between the coupled fluidized bed reactors is controlled by a screw conveyor. In order to reach realistic oxy-fuel conditions, the calciner is fired with oxygen enriched recirculated flue gas. The heat release of the carbonation reaction can be extracted directly from the carbonator bed by means of five axially arranged internal cooling tubes. The flue gases are cooled down by means of two-pass heat exchangers. The entrained solid particles are removed from flue gases in bag filters.

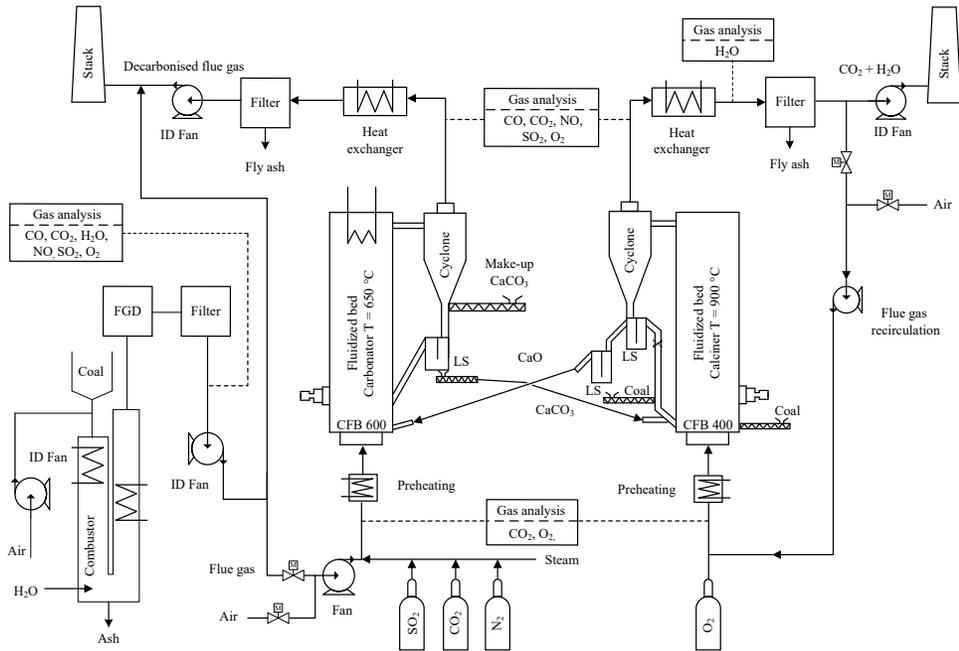


Fig. 1. Scheme of upgraded 1 MW_{th} CaL pilot plant at Technische Universität Darmstadt.

The inventory of solids in the carbonator and calciner is continuously determined by the measurement of the reactor pressure drop between the plane above the distributor and the exit of the reactor. The average composition of the solids entering and leaving the carbonator leaving is frequently determined from solid sampling using sampling ports in the loop seals and the screw conveyor. Also samples from the heat exchanger and the filters are taken. Chemical analysis of solids samples is performed after test campaigns. Thus, it was possible to define the molar carbonate content of solids leaving the carbonator (X_{carb}) and the calciner (X_{calc}) as well as to the deactivation caused by sintering and enrichment of impurities (ash, calcium sulphate). The solid mass flow circulating between the reactors can be calculated by the volumetric characteristic curve of the screw conveyor applying the solid composition determined by solid analysis. Gas compositions and volumetric flows of the in- and outgoing gas streams are measured. At the carbonator inlet, the CO_2 , O_2 , NO , CO , SO_2 and H_2O concentration of flue gas is measured, as well as the volumetric flow of flue gas. Additional CO_2 and O_2 concentration measurement in the primary gas line are installed. In the calciner, O_2 and CO_2 concentration, and the volumetric flow at the inlet are measured. Outlet concentrations CO , CO_2 , NO_x , SO_2 and O_2 as well as volumetric flows of both reactors are measured. Additionally, H_2O concentration at calciner outlet is measured. Measurements of flow rates, pressure, temperature, concentration, gas compositions, solids loading and velocity at numerous locations were carried out to evaluate and characterize the overall process.

The trends observed during operation, especially in steady-state periods, are evaluated in terms of the efficiency of the process. The CO_2 absorption efficiency in the carbonator E_{carb} is crucial parameter for process evaluation in terms of reactor design and scale-up purposes. It is defined as the flow of CO_2 captured from the CO_2 fed in the carbonator (see Fig. 3a). The carbonator efficiency can be calculated as follows:

$$E_{\text{carb}} = \frac{F_{\text{CO}_2, \text{carb}, \text{in}} - F_{\text{CO}_2, \text{carb}, \text{out}}}{F_{\text{CO}_2, \text{carb}, \text{in}}} \quad (2)$$

The maximum carbonator efficiency for removing CO_2 from the gas flow entering the carbonator is limited by the minimum CO_2 fraction given by the chemical equilibrium. The chemical equilibrium is a function of the average carbonator temperature T_{carb} and is shown in Eq. (3) from Silcox et al. (1989):

$$v_{\text{CO}_2, \text{eq}} = 4.137 \times 10^7 \exp\left(\frac{-20,474}{T}\right) \quad (3)$$

The total CO_2 capture efficiency E_{total} of the CaL process is defined as the relationship between the captured CO_2 at the calciner outlet ($F_{\text{CO}_2, \text{calc}, \text{out}}$) and produced CO_2 . The total amount of produced CO_2 includes the CO_2 fed from the host plant ($F_{\text{CO}_2, \text{carb}, \text{in}}$), CO_2 released by calcination from fresh limestone ($F_{\text{CO}_2, 0}$) as well as CO_2 formed by combustion of coal in the calciner ($F_{\text{CO}_2, \text{coal}}$), shown in Eq. (4).

$$E_{\text{total}} = \frac{F_{\text{CO}_2, \text{calc}, \text{out}}}{F_{\text{CO}_2, \text{carb}, \text{in}} + F_{\text{CO}_2, 0} + F_{\text{CO}_2, \text{coal}}} \quad (4)$$

Four comprehensive long-term CaL test campaigns of four weeks each were conducted. Test campaigns #1 and #3 were performed with hard coal either in pulverized ($d_{50} \sim 36\text{--}45 \mu\text{m}$), coarser pulverized ($d_{50} \sim 500 \mu\text{m}$) and coarse form ($d_{50} \sim 1500 \mu\text{m}$), test campaigns #2 and #4 with pulverized ($d_{50} \sim 35 \mu\text{m}$) or grained lignite ($d_{50} \sim 500 \mu\text{m}$), respectively. During these test campaigns, two different sorbents from western (LimestoneA, $d_{50} \sim 180 \mu\text{m}$) and southern (LimestoneB, $d_{50} \sim 215 \mu\text{m}$) Germany with different chemical compositions and particle sized distributions were utilized. Table 1 presents an overview of the test campaigns including the number of test campaigns, performed between September 2015 and April 2016, the operating conditions and the fuel of the calciner and the sorbent used for the tests.

Table 1. Overview of the CaL test campaigns.

	Calciner fuel	Calciner fuel size		Calciner mode	Sorbent
1	hard coal	coarse	$d_{50} \sim 1500 \mu\text{m}$	oxy-fuel fired	LimestoneA
	hard coal	pulverized	$d_{50} \sim 45 \mu\text{m}$	air / oxy-fuel fired	LimestoneA
2	lignite	pulverized	$d_{50} \sim 35 \mu\text{m}$	oxy-fuel fired	LimestoneA
3	hard coal	pulverized	$d_{50} \sim 36 \mu\text{m}$	oxy-fuel fired	LimestoneB
	hard coal	coarser pulverized	$d_{50} \sim 500 \mu\text{m}$	oxy-fuel fired	LimestoneB
4	lignite	pulverized	$d_{50} \sim 35 \mu\text{m}$	oxy-fuel fired	LimestoneA
	lignite	pulverized	$d_{50} \sim 35 \mu\text{m}$	oxy-fuel fired	LimestoneB
	lignite	grained	$d_{50} \sim 500 \mu\text{m}$	oxy-fuel fired	LimestoneB

The detailed evaluation including heat and mass balances as well as detailed sorbent analysis aims to gain a deeper understanding of the CaL process with a distinguished focus to apply the experimental data for scaling-up the CaL process to a realistic demonstration plant.

RESULTS AND DISCUSSION

Steady-state and transition periods from long-term pilot testing are exemplarily shown in Fig. 2. Generally, an experimental test campaign starts with heating of the reactors by start-up burners and coal, calcination of the solid inventory and start of flue gas recirculation in the calciner. Then operating conditions, e.g. make-up flow, coal feed, solid circulation etc. are adapted to adjust the operating conditions. Sorbent activity needs to be adjusted as well as loss of fines due to attrition has to be replaced by make-up addition.

Exemplary results from a CaL long-term test campaign in the 1 MW_{th} pilot plant of TU Darmstadt are shown in Fig. 2 a-c. In the initial period until hour 35 (1), the CaL process was started. Temperatures T_{calc} of 900 °C in the calciner and T_{carb} of 650 °C in the carbonator were adjusted (Fig. 2b). The reactor velocities u_{carb} and u_{calc} were kept around 5 m/s in the calciner and 3 m/s in the carbonator, respectively (Fig. 2b). The specific carbonator inventory $W_{\text{s,carb}}$ was 500 kg/m² (Fig. 2c). The solid circulation rate between the reactors G_{s} was increased to 3.9 kg/m²s (Fig. 2c) and the make-up flow rate F_0 was increased from 0.18 to 0.38 mol/m²s (Fig. 2a) with the aim to achieve a carbonator absorption rate E_{carb} higher 80 % (Fig. 2a).

In the period from hour 35 (1) to hour 53 (2), CO₂ was continuously captured in the set range of parameters. The flow of CO₂ captured in the carbonator $F_{\text{CO}_2,\text{carb,in}} \times E_{\text{carb}}$ increased up to 4.1 mol/m²s leading consecutively to E_{carb} higher 80% (Fig. 2a).

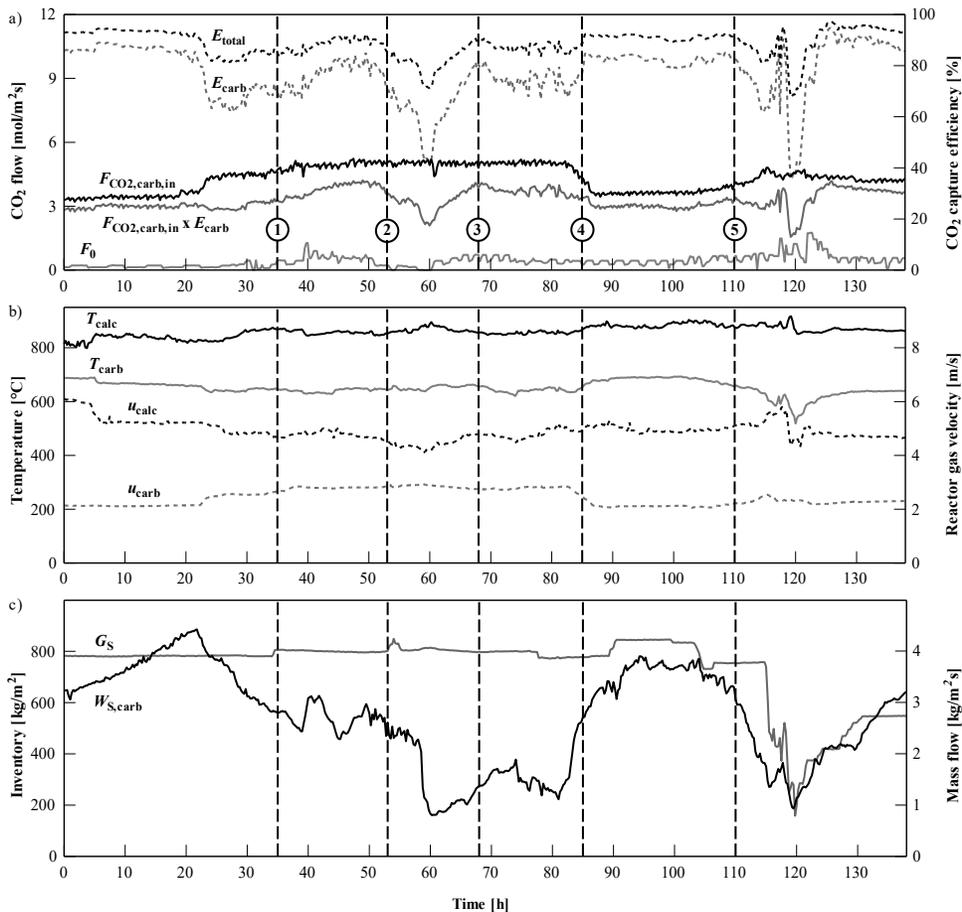


Fig. 2. Exemplary period of long-term CaL tests.

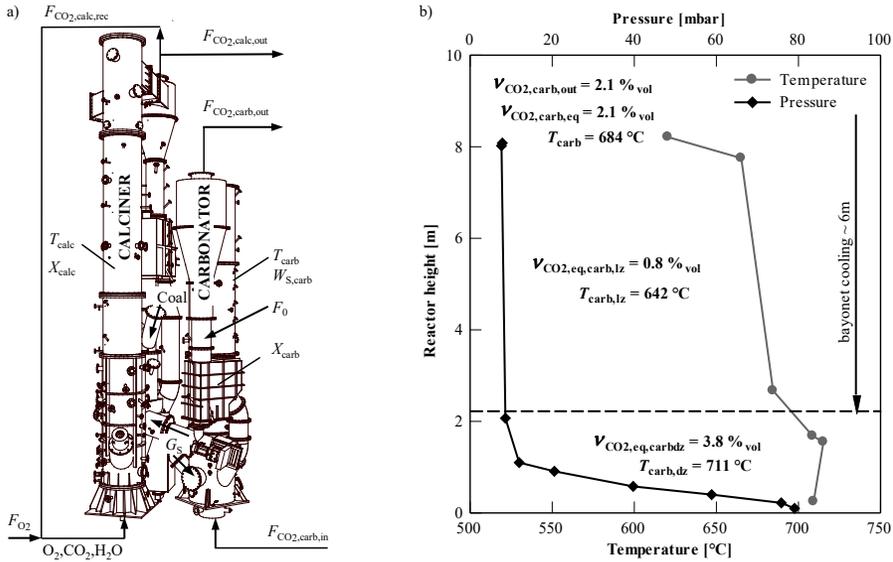


Fig. 3: Schematics with the main molar flows and operating parameters of the pilot plant (a) and exemplary carbonator temperature and pressure profile during period 4-5 (b).

The continuous operation was interrupted at hour 53 (2). Blockings in the carbonator cyclone led to a discontinuous internal solid circulation in the system and inventory was discharged via cyclone. As a consequence, $W_{S, carb}$ decreased to 500 to 300 kg/m² and E_{carb} dropped to 40 % (see Fig. 2c).

Stable operation was reestablished at hour 68 (3). The operation with previous conditions was continued except the solid inventory in the carbonator. E_{carb} was kept at a high level with the lower inventory since G_S (see Fig. 2c) and F_0 (see Fig. 2b) were kept constant. Some fluctuation in the flow of captured CO₂ and consequently in the carbonator efficiency can be observed. Since the CO₂ capture reaction in the carbonator in this period was kinetically controlled, fluctuations in the solid flow entering the carbonator caused fluctuations in the carbonator inventory as well as the flow of captured CO₂ and subsequently in the carbonator absorption rate.

The testing period was finished at hour 85 (4) and a new test point was adjusted. The carbonator was refilled, the incoming CO₂ flow was decreased and the solid circulation rate was increased in order to switch from kinetically to chemical equilibrium controlled absorption in the carbonator. As a side effect, carbonator temperature was slightly increased by the amount of additional solid entering the carbonator. During this period, a very stable operation could be accomplished and only very little fluctuations in the CO₂ flow captured in the carbonator and subsequently in the carbonator absorption efficiency were observed for a period of 20 hours. Minor parameter variations were tested until the end of this operation scenario after hour 110 (5).

An exemplary temperature and pressure profile of the carbonator reactor during this period is shown in Fig. 3b. The hydrodynamic pressure profile indicates the solid distribution in the reactor. A distinct dense zone in the lower part of the reactor is shown by the pressure profile. The dense zone contained most of the inventory whereas only a minor part of the inventory was in the lean zone. In this period, the cooling was accomplished by bayonet cooling tubes in the carbonator to control the average reactor temperature T_{carb} to 684 °C leading to a volumetric equilibrium concentration $v_{CO_2, carb, eq}$ of 2.1 %vol. The temperature profile shows a distribution between a warmer dense zone ($T_{carb, dz} = 711\text{ °C}$) and a colder lean zone ($T_{carb, lz} = 642\text{ °C}$). In-bed gas analysis show that the main part of the CO₂ is absorbed in the dense zone of the reactor (>80% of E_{carb}). Equilibrium conditions $v_{CO_2, eq, dz}$ of 3.8 %vol could be achieved in the dense zone. The carbonation reaction runs faster since the higher dense zone temperatures positively affect the reaction kinetics. In order to achieve high carbonator absorption efficiencies, the temperature in the lean zone is lowered by the cooling tubes. There, equilibrium conditions $v_{CO_2, eq, lz}$ of 0.8 %vol are predominant and carbonation reaction can continue. Approx. 15% of the total carbonator absorption efficiency is realized in the lean zone. All in all following this strategy, high carbonator absorption efficiencies higher than 80 % at equilibrium conditions in the reactor ($v_{CO_2, carb, out} = 2.1\% \text{ vol}$) could be achieved. Equilibrium limited carbonator operation shows a very stable behaviour during the total operating period.

Table 2: Composition and particle size of various calciner fuels during pilot operation.

		hard coal	lignite
		coarse / pulv.	pulv.
d_{50}	μm	1,500 / 45	35
LHV	MJ/kg	26.5	22.2
Sulphur	kg/MJ	0.026	0.014
Ash	kg/MJ	0.587	0.166

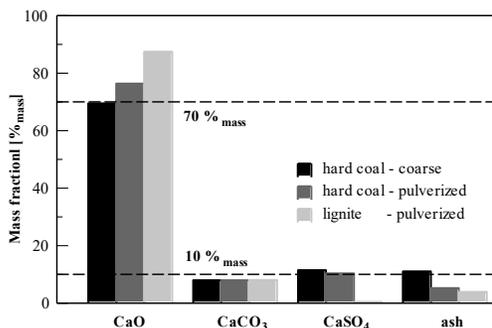


Fig. 4. Solid composition of circulating solid flow between reactors with the same make-up rate ($\sim 0.7\%$ mol_{Ca}/mol_{Ca}).

Long-term pilot operation was carried out with various types of fuels. Fuel properties as well as particle sizes were varied. Table 2 provides an overview of the coal used for flue gas production in the furnace as well as providing the required heat for calcination in the calciner reactor. Colombian hard coal was used either in coarse ($d_{50} \sim 1,500\ \mu\text{m}$) or in pulverized ($d_{50} \sim 45\ \mu\text{m}$) particle size in the calciner with a lower heating value (LHV) of 26.5 MJ/kg. Fig. 4 shows the effect of the fuel type and particle size on the sorbent circulating during CaL pilot tests with the same feed ratio of make-up. Obviously, significant difference in the amount of inactive material (gypsum, ash) can be observed depending both on the particle size and on the type of fuel. Using hard coal with the same composition and different particle size distribution, it can be observed that the share of gypsum is similar ($\sim 10\%$ mass) and ash differs. With coarse hard coal, 11 % mass of ash accumulates in the circulating solid stream compared to 5 % mass firing pulverized coal. The particle size of the coal feed influences the ash separation efficiency in the calciner cyclone. The bigger the ash particles, the more ash is separated and accumulates in the circulating material. A significant difference in gypsum amount can be recognized comparing the composition of the samples for pulverized hard coal as well as lignite. A rather low share of 0.5 % mass gypsum accumulates in solid circulation firing lignite compared to 10 % mass with hard coal. The low sulphur content of the lignite compared to the hard coal (see Table 2) has a positive effect on accumulation of inert material in the process. The sulphur input from the flue gas from the combustor as well as in the calciner firing was lower compared to the hard coal tests.

CONCLUSION

The CaL technology is one of the most promising post-combustion CCS technologies. An important step towards the realization of this technology in commercial scale is the successful pilot testing in 1 MW_{th} scale. Semi-industrial pilot testing of the CaL process during 16 weeks with more than 1,200 hours of stable CO₂ capture showed the maturity of the process realizing high CO₂ capture rates in steady-state operation. During steady-state operation, CO₂ absorption rates in the carbonator higher than 90 % and overall CO₂ capture rates higher than 95 % were proven under a wide range of parameters, e.g fuel characteristics in the calciner, solid circulation rates, make-up rate etc. The calciner fuel particle size and the fuel composition significantly influence the composition of the sorbent circulation between the reactors and thus, its performance to absorb CO₂ from the flue gas lead in the carbonator. Enrichment of impurities, meaning ash and sulphur in the circulating solid, are mainly depending on the composition of the coal fueled in the calciner. Increased amounts of impurities represent an inactive fraction decreasing the ability of the sorbent of absorbing CO₂. This effect can be compensated by either increased addition of fresh material and extraction of used material or raising the circulating solid stream between the reactors. The data provide the basis for scale up of the CaL technology to a CCS demonstration plant in 20 MW_{th} scale. Based on the conclusions drawn from operating experience, heat and mass balancing as well as solid analysis are used for design and engineering of a 20 MW_{th} CaL pilot plant as the next step towards maturity of the technology.

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NOTATION

Abbreviations

CaL	Calcium Carbonate Looping
CCS	Carbon Capture and Sequestration
CFB	Circulating Fluidized Bed
EST	Institute for Energy Systems a. Techn.
IGCC	Int. Gasification Combined Cycle
IHCaL	Indirectly heated CaL
LHV	Lower Heating Value
MEA	Mono-Ethylen-Amine

Nomenclature

d_{50}	median particle diameter, μm
E_{carb}	carbonator absorption efficiency
E_{total}	total process capture efficiency
F_0	make-up flow, $\text{mol}/\text{m}^2\text{s}$
$F_{\text{CO}_2,0}$	CO_2 flow released from make-up, $\text{mol}/\text{m}^2\text{s}$
$F_{\text{CO}_2,\text{carb,out}}$	CO_2 flow leaving the carbonator, $\text{mol}/\text{m}^2\text{s}$
$F_{\text{CO}_2,\text{carb,in}}$	CO_2 flow entering the carbonator, $\text{mol}/\text{m}^2\text{s}$
$F_{\text{CO}_2,\text{calc,out}}$	CO_2 flow leaving the process, $\text{mol}/\text{m}^2\text{s}$

$F_{\text{CO}_2,\text{coal}}$	CO_2 flow from coal combustion, $\text{mol}/\text{m}^2\text{s}$
G_s	solid flow rate, $\text{kg}/\text{m}^2\text{s}$
T	temperature, $^\circ\text{C}$
T_{calc}	average calciner temperature, $^\circ\text{C}$
T_{carb}	average carbonator temperature, $^\circ\text{C}$
$T_{\text{carb,dz}}$	carbonator dense zone temperature, $^\circ\text{C}$
$T_{\text{carb,lz}}$	carbonator lean zone temperature, $^\circ\text{C}$
u_{calc}	calciner gas velocity, m/s
u_{carb}	carbonator gas velocity, m/s
$W_{\text{S,carb}}$	total carbonator inventory, kg/m^2
X_{calc}	carbonate content of calciner solids
X_{carb}	carbonate content of carbonator solids

Greek letters

$v_{\text{CO}_2,\text{eq}}$	CO_2 equilibrium (eq.), $\%_{\text{vol}}$
$v_{\text{CO}_2,\text{eq,carb}}$	CO_2 eq. carbonator, $\%_{\text{vol}}$
$v_{\text{CO}_2,\text{eq,carb,dz}}$	CO_2 eq. carbonator dense zone, $\%_{\text{vol}}$
$v_{\text{CO}_2,\text{eq,carb,lz}}$	CO_2 eq. carbonator lean zone, $\%_{\text{vol}}$
$v_{\text{CO}_2,\text{carb,out}}$	CO_2 concentration carbonator outlet, $\%_{\text{vol}}$

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