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CHEERS demonstration unit operational results for the European configuration

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Abstract

The CHEERS (Chinese European Emission Reduction Solution) project is an international consortium aiming at semi-industrial demonstration of the Chemical Looping Combustion (CLC) technology at 3 to 5 MW_{th} scale. The nine partners of the project are SINTEF Energy AS, TotalEnergies, IFP Energies Nouvelles, Bellona, SINTEF AS, Silesian University and Dongfang Boiler Group Co (DBC), Tsinghua University and Zhejiang University. The demonstration plant was built on the site of DBC in Deyang China in 2022. The first operational test phase in unit configuration 1 (EU) were conducted between June 2023 and March 2024. Configuration 2 (CN) operational test followed this first phase.
This paper presents the main outcomes of the last phases of the project: start-up, CLC mode setup and operational tests in CLC mode. Before the start-up phase, a supplemental phase consisting of pre-oxidizing the ilmenite used as oxygen carrier is described. The start-up phase consists of heating up the unit and obtaining circulation of oxygen carrier between the two reactors. After the start-up phase, stable continuous operation in autothermal CLC mode was achieved, demonstrating the reliability of the developed CLC process. The main key performance indicators, carbon capture efficiency and oxygen demand of the unit for the two feedstocks tested, lignite and grinded petcoke, where found to be up to 94% carbon capture efficiency and as low as 2.5% oxygen demand. The best results were achieved for lignite. When compared to other CLC units in the world, the performance of the CHEERS demonstration unit is found to be very good.

Keywords: CLC; CHEERS; Pilot operation; Results, Autothermal

1. Introduction

Chemical Looping Combustion (CLC) is a second-generation CO_2 capture technology. Techno-economic studies have shown that it has the potential to provide significant reduction in both efficiency penalty and CO_2 avoidance cost when capturing CO_2 from conversion of solid fuels, such as coal and biomass. In these techno-economic studies, CLC has been benchmarked against reference processes, mainly amine-based post-combustion CO_2 capture and oxy-combustion CO_2 capture processes. A more recent public study by the CHEERS project clearly underpins these earlier results, that CLC with solid fuels can achieve clear benefits with respect to efficiency and costs.
To progress the technology further up in TRL, operational experience and performance must be obtained at scales larger than what has been used so far. The divided combustion process of a CLC unit is more complex than a standard CFB process. The process performance is very dependent on the properties of the oxygen carrier material (OC). The energy balance and reactor temperatures are closely coupled to the OC circulation. Close control of OC circulation, OC inventories, and pressure balance of the loop is required. The process must be designed to match these operational requirements, plus providing low heat losses and safe operation in all phases. All these aspects become even more important at a larger scale, closer to the industrial case.
Therefore, the overall goal of the CHEERS project was to advance CLC from a technology readiness level (TRL) of 5 up to TRL 7 by the end of the project. This should be done by designing and building the world's largest CLC unit and perform tests and assess key performance indicators at a more industrially relevant scale.
The commissioning of the CHEERS demonstration unit was performed in the second quarter of 2023. After testing all the unit's equipment individually and validating all the safety conditions, a permit to start was delivered and the unit could enter the operation phase. The unit start-up was done on 6 June 2023 and the operational test of the current configuration 1 (EU) lasted until the end of March 2024.

Nomenclature

AR	Air Reactor
CCE	Carbon Capture Efficiency
CEMS	Continuous Emission Monitoring System
CFB	Circulating Fluidized Bed
CLC	Chemical Looping Combustion
CN	Chinese
CS	Carbon Stripper
DBC (DONGFANG)	Dongfang Boiler Group Co., LTD
DFB	Divided Fluidized Bed
EU	European
FR	Fuel Reactor
IFPEN	IFP Energies Nouvelles
LV	L-Valve
LHV	Low Heating Value
MFB-TGA	Micro-fluidized bed thermogravimetric analyser
MW	Mega Watt
OC	Oxygen Carrier
OD	Oxygen Demand
PSD	Particle Size Distribution
Re	Reynolds Number
TGA	Thermogravimetric analyser

2. CHEERS demonstration unit start up preparation

2.1. Feedstock

This chemical looping combustion process use solid fuels, including but not limited to lignite, petcoke, and biomass. Among these, lignite has undergone extensive testing and is the most widely studied solid fuel for the CLC process. Lignite is distinguished by its rapid char gasification rate and high volatile content, making it advantageous for carbon capture efficiency as the carbon from the volatiles is consistently captured in the fuel reactor. In contrast, petcoke is characterized by low volatile matter and low char reactivity, necessitating a highly efficient carbon stripper to separate unconverted char particles for their return to the fuel reactor to achieve optimal CO_2 capture efficiencies.
The fuel used in the CHEERS demonstration unit is either lignite or petcoke. The lignite used is sourced from Indonesia. The solid fuel feed (petcoke or lignite) compositions are given in Table 1.

Table 1: Typical solid fuel composition

Parameter	Unit	Lignite	Petcoke
Moisture	wt. %	35.29	0.63
Fixed Carbon	wt. %	26.50	90.89
Volatile Matter	wt. %	34.60	8.32
Ash	wt. %	3.61	0.16
C	wt. %	45.31	94.24
H	wt. %	3.35	1.33
N	wt. %	0.27	1.76
S	wt. %	0.08	0.86
O	wt. %	12.09	1.02
LHV	kJ/kg	15890	33565

2.2. Oxygen Carrier: Ilmenite

Many different synthetic oxygen carriers and minerals were tested upfront of the CHEERS demonstration unit operational test. After tests for reactivity and kinetics towards fuel in FR and air in AR, oxygen transfer capacity, chemical and mechanical strength, attrition, poisoning components tolerance, cost and disposal, the most suitable oxygen carrier for operational test was selected. Ilmenite delivered by Titania in Norway was selected for the CLC test. The main characteristics are shown in Table 2.

Ilmenite is a common mineral found in metamorphic and igneous rocks, mainly composed of $FeTiO_3$ where iron oxide is the active phase that behaves as oxygen carrier. In principle, when using ilmenite as an OC in a CLC process, the reduced form consists of $FeTiO_3$ $(FeO \cdot TiO_2)$ and the most oxidized level is Fe_2TiO_5 $(Fe_2O_3 \cdot TiO_2)$.

Table 2 : Properties of ilmenite used in calculation.

Items	Unit	value
Oxygen transport capacity R_0	%	5.30
Specific heat capacity	kJ/K/kg	0.91
Fresh Particle density	kg/m^3	4800
Activated particle density	kg/m^3	2500
Bulk density	kg/m^3	1437

It has been demonstrated by different studies in the literature that ilmenite at its natural state (fresh ilmenite) is neither at the maximal oxidative state nor at the maximum reactivity capacity. To obtain an ilmenite suitable for CLC technology at an adequate oxygen transport capacity, ilmenite needs to follow two preparation stages: pre-oxidation and activation.

The first stage is the pre-oxidation or calcination of ilmenite to bring this solid to the most oxidative state. This step is important to improve the fluidisation of the OC and to avoid agglomeration issues in the static zones of the unit (for example, L-valves or loop seals). To oxidate ilmenite, it is heated in presence of oxygen (air) at a temperature higher than 800°C. 75 tonnes of ilmenite were pre-oxidized in CHEERS unit using a dedicated protocol.

The second stage consists in activating ilmenite to obtain the maximum reactivity capacity. Ilmenite is one of the oxygen carriers that need to follow various redox cycles to be activated and successfully used in CLC process. Initially, ilmenite reacts slowly. After a certain number of cycles, there is a gain in the capacity to be reduced and oxidized due to the openness of the porosity. Increase of the porosity is consequently a good sign of particles activation in the process. By opening the porosity of the particles, the mechanical resistance will decrease and generate more attrition and fines particles.

3. CHEERS demonstration unit start-up procedure

3.1. CHEERS demonstration unit description

The *Figure 1* below describes the main equipment of the unit. The DFB (Divided Fluidized Bed) is an oxygen carrier buffer to be able to either recirculate oxidised OC to the AR if the oxidation step in one pass is not sufficient, or to go directly to the FR bottom to transport oxygen for the fuel combustion.

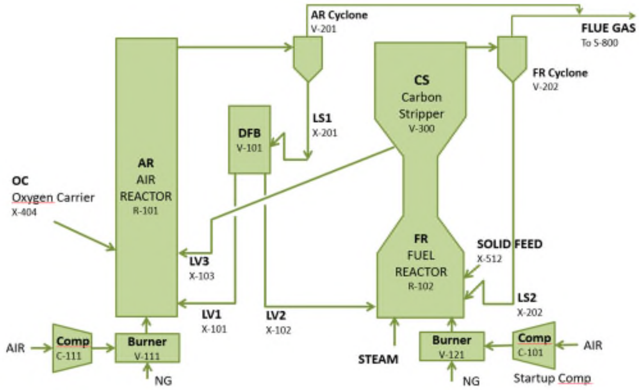


Figure 1. CLC CHEERS demo unit main equipments.

3.2. From cold condition to OC circulation

<u>Main steps to go to CLC mode</u>

Achieving Chemical looping combustion in CHEERS unit requires going through several stages to do the heating of the unit gradually and reach optimal operating conditions needed for the reduction and oxidation of the ilmenite in the fuel and air reactor and successful CLC operation:

1. Heating up the unit to 950°C with natural gas (NG), then lignite.
2. Switching from lignite to petcoke.
3. Maintaining a stable circulation of solid at a flowrate above 50 t/h.
4. Switching the fluidisation gas from air to steam in the capacities: DFB, CS, X-201 and X-202.
5. Switching the fluidisation gas from air to steam in the fuel reactor.
6. Activation of the ilmenite through various cycles of oxidation/reduction to open ilmenite porosity and make active sites accessible.

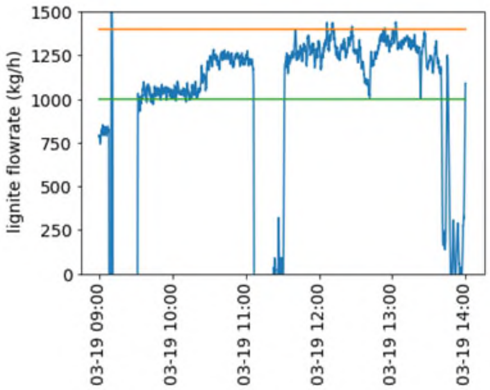
<u>Injection system</u>

Lignite powder is transported to the FR via adjustable frequency rotary valve and a screw. Grinded petocke powder is injected via adjustable frequency rotary valve followed by a pneumatic injection at 5 and then 20 m/s to obtain a deep penetration of petcoke in the fluidized bed.

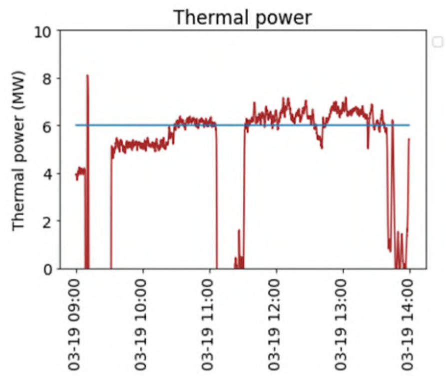
Ilmenite (OC) is injected via adjustable frequency rotary valve in the AR. All rotary valves had been calibrated prior start-up.

<u>Lignite feeding calculation and Power estimation</u>

Lignite feeding flowrate measured by the weighting system is shown in Figure 2(a). We find that lignite flowrate is between 1000 and 1400 kg/h (marked by two continuous lines in the Figure). Note that the strong fluctuation on the curve (when the curve drops to zero) corresponds to the recharge of the lignite silo. Thus, values of strong fluctuation should not be considered to estimate lignite feeding flowrate. The mean lignite feeding flowrate is then **1209 kg/h**. Knowing that the calorific value of lignite is about 17.9 MJ/kg, the corresponding thermal power of the unit is about **6MW** Figure 2(b).



(a)



(b)

Figure 2: lignite feeding flowrate given by the weighting system installed under lignite silo.

4. CLC operational results

4.1. KPI: Key Performance Indicator definition

KPI 1: Carbon Capture Efficiency

One of the key parameters to evaluate the quality of the CLC mode is the Carbon Capture Efficiency (CCE or η_{CC}). It estimates the percentage of the carbon going out of the FR (which will be captured) over the total carbon going out of the unit (considering the char going in the AR with the OC). Consequently, the CCE is directly linked to the Carbon Stripper efficiency (char sent to the AR). The following equation is used [1]:

$$CCE \ or \ \eta_{CC} \ (\%) = \frac{F_{CO2}^{FR,out} + F_{CO}^{FR,out} + F_{HC}^{FR,out} - F_{CO2}^{FR,in}}{F_{CO2}^{FR,out} + F_{CO}^{FR,out} + F_{HC}^{FR,out} - F_{CO2}^{FR,in} + F_{CO2}^{AR,out}} \quad (1)$$

with F_x the molar flowrate (mol/s) of the species x (at inlet/outlet of FR/AR). As an example, $F_{CO2}^{FR,in}$ corresponds to the CO_2 molar flowrate injected in the FR (L-valve aeration, pressure drops insufflation, fuel injection line…). This term is removed from the above equation because this is not carbon produced by the reaction.

Another way to evaluate the Carbon Capture Efficiency is to calculate what is called the “oxide oxygen fraction (χ_{OO})”. This number is defined as the amount of oxygen used for oxidizing the OC in the air reactor divided by the sum of that used for oxidizing both char and OC in the air reactor. [1]. The advantage of this method is that it does not require gas concentration measured in FR and eliminate any uncertainties due to flows. The following equation is used:

$$\chi_{OO}(\%) = \frac{0.21 - y_{O2,AR} - y_{CO2,AR}}{0.21 - y_{O2,AR} - 0.21 * y_{CO2,AR}} \quad (2)$$

With $y_{X,AR}$ the molar fraction of the species x (at the outlet of AR). As an example, $y_{O2,AR}$,AR corresponds to the O_2 molar fraction measured at the AR outlet.

KPI 2: Oxygen Demand

Another key parameter to evaluate the global efficiency of the quality of the CLC mode is the total Oxygen Demand (Ω_T). The oxygen demand is the fraction of oxygen needed for the combustion of all the unburned compounds produced and the oxygen needed for the complete combustion of the introduced solid fuel [1].
The following equation is used to calculate Ω_T:

$$\Omega_T\ (\%) = \frac{4*F_{CH4}^{FR,out} + F_{CO}^{FR,out} + F_{H2}^{FR,out}}{\frac{1}{M_O}*\Omega_{SF}*m_{SF}} \quad (3)$$

- F_x the molar flowrate (mol/s) of the species x
- M_O the molar mass of oxygen (kg/mol)
- Ω_{SF} oxygen demand of the solid fuel (kg oxygen per kg solid fuel)
- m_{SF} mass flow of solid fuel fed in FR (kg/s)

It is also possible to calculate an Oxygen demand in the FR (Ω_{FR}) with respect to the amount required to burn the solid fuel converted in the fuel reactor [1]. The formula is as follows:

$$\Omega_{FR}\ (\%) = \frac{4*F_{CH4}^{FR,out} + F_{CO}^{FR,out} + F_{H2}^{FR,out}}{\frac{1}{M_O}*\Omega_{SF}*m_{SF} - 2*F_{CO2}^{AR,out} - 2*F_{C,elut}} \quad (4)$$

- $F_{CO2}^{AR,out}$: molar flowrate (mol/s) of CO_2 at AR outlet
- $F_{C,elut}$: carbon flow in the char elutriated in the CS (mol/s)

The term $F_{C,elut}$ can be calculated by making a hypothesis on the char conversion in the FR and saying that the char is only composed of carbon. The value for the char conversion has been taken equal to 75% for lignite feedstock and 20% for petcoke feedstock.

4.2. Lignite CLC results

Autothermal CLC for Lignite feedstock injection

For the first CLC test in March 2024, the switch air/steam was initiated on 17th March and the FR was completely subjected to steam in the evening the same day. The unit was at first in autothermal mode with a mix feedstock of Lignite and not grinded petcoke and secondly with a pure lignite feedstock. The results in this section are the one with the Lignite feedstock based on data recorded on the 19th March.
Reaching autothermal mode is key for the applicability of the CLC process. For a CLC process to be defined as autothermal, it is a requirement that there is no injection of fuel elsewhere than FR, and that steam is the only fluidization agent in the FR. In addition, there can be no external heat sources acting on the system. In that configuration, the heat generated in the AR by OC reoxidation maintain the temperature in the unit with the solid circulation (compensation of thermal losses).
It is important to mention that the temperature in the FR is directly linked to the OC circulation rate, or, more specifically, to the OC flowrate entering the FR. Because of high heat losses due to refractory degradation, a high solid circulation rate and a high thermal input was needed to keep the temperature around 900 °C in the FR. OC circulation flowrate was calculated from a model based on AR delta Pressure drop related to the solid particle weight, acceleration, and friction. [2]

Results Lignite KPI 1

Independently of which way is selected, the parameter needs to be evaluated in steady state operating conditions under CLC mode (meaning no fuel injected in AR). The conditions are shown below in Table 3.

Table 3: Steady state operating conditions during CLC mode with lignite as fuel

FR temp (°C)	AR top temp (°C)	Fuel flowrate (t/hr)	MW	OC flowrate (t/hr)	CO_2 AR outlet (%v)	CO_2 FR outlet (%v)
900	1000	1.1	5	145	2.5-3*	96.5*

*Note that X-102 and X-103 L-valves are fluidized with CO2 and influence the CO2 amount at AR and FR outlet. This fluidization stream flowrate measured had been subtracted for KPI calculation.

However, for the Carbon Capture calculations, those amounts calculated before CLC mode are subtracted.

The gas concentration at FR outlet (CO, CO_2, HC and H_2) given by the CEMS analyser encountered a lot of issues. Consequently, the gas concentration is not reliable. To calculate the CCE (KPI 1) and Oxygen Demand (KPI 2), external gas analysis (DBC's laboratory in Chengdu) of gas sampling were used.
Figure 3 shows the results of the calculation for carbon capture efficiency under steady state conditions for both ways explained below (CCE and χ_{OO}). The mean value under this period for both methods is 91%. This is a promising value since lignite particle size distribution is high and consequently CS efficiency is lower than expected (the CS design was based on a petcoke particle size distribution that is lower than the current lignite size distribution). However, by definition, CCE is higher for fuel with high volatiles content (and consequently low char content) such as lignite or biomass. Both ways to calculate CCE give similar results and have the same trend.

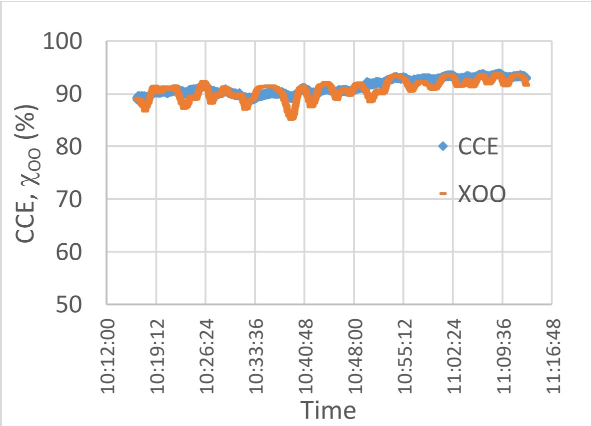


Figure 3: Estimation of the carbon capture efficiency under steady sate operating conditions with lignite as fuel (19/03/2024)

<u>Results Lignite KPI 2</u>

The equations used to calculate Total Oxygen Demand KPI 2 can be found in section 4.1.
The term $F_{C,elut}$ can be calculated by making a hypothesis on the char conversion in the FR. The value has been taken equal to 75 % for lignite feedstock.
Figure 4 shows the results of the calculation for total Oxygen Demand and Oxygen Demand in FR under steady state conditions. The mean values under this period are respectively 2.26 % and 2.61 %. The value for the Oxygen Demand (Total and in FR) is quite stable because gas composition is taken from external gas analysis.

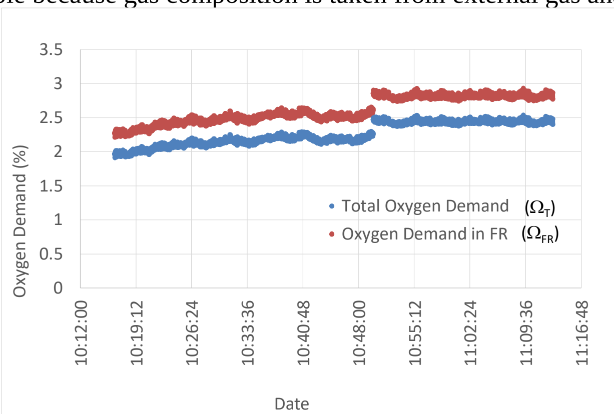


Figure 4: Estimation of the oxygen demand evolution under steady sate operating conditions with lignite as fuel (19/03/2024)

The mean values for "Total Oxygen Demand" and "Oxygen Demand in FR" during CLC mode with lignite are very low compared to literature data [1].
Since we know that the carbon capture efficiency is high, this means that the combustion efficiency is very good. High

solid circulation rate (consequently high oxygen to fuel ratio) coupled with good hydrodynamic conditions in FR can explain such a low value. This is a positive sign for the FR bottom design which allows, even with high solid fuel flowrate, a very good combustion efficiency. Furthermore, achieving low oxygen demand for fuels with high volatiles content, such as lignite or biomass, is challenging since the high volatile content tend to release gases more readily when entering the reactor.
Based on these latest results for high CCE and low Oxygen Demand for lignite in CLC mode, it is possible to say that the CLC efficiency was very good with lignite as fuel. Note that the very high solid circulation rate is one of the reasons for such a good CLC efficiency, since it ensures a good combustion of the volatiles generated (high oxygen to fuel ratio).

Gas sampling at AR outlet

For the gas leaving the AR, O_2 and CO_2 concentrations given by CEMS. To compare the data given by O_2 and CO_2 analysers, one gas sampling analysis has been performed at the AR outlet and analysed in an external lab. Results are shown below in *Table 4*:

Table 4: Gas analysis results for AR outlet gas sampling during CLC mode with lignite as fuel

Sample	O_2 (%v)	CO_2 (%v)
19/03/2024 10h	10.4	3.5

CO_2 concentration given by gas sampling analysis seems close to the value given by the CO_2 analyser. Otherwise, even if the O_2 concentration is bumpy, the O_2 concentration given by gas sampling analysis is not totally consistent: 10.4 % versus 7.5% (mean value). Note that this is a single spot value and so it is difficult to compare with online measurements.

Gas analyses at FR outlet

Results of FR outlet dry gas analyses are shown in *Table 5*. This sample was taken on March 29th at 9:39 (lignite as fuel). The values listed in *Table 5* have been taken when estimating KPIs.

Table 5: Gas analysis results for FR outlet gas sampling during CLC mode with lignite as fuel

Component	Content
CO_2	96.5 %vol
CO	1.6 %vol
H_2	1.3 %vol
CH_4	0.5 %vol

Ilmenite $R_0\Delta x$ analysis

During test campaign, ilmenite was sampled in DFB for the oxidized phase and in the FR for the reduced phase. It was then possible to measure the $R_0\Delta x$ of the Ilmenite. Two methods were used. A delta weight after and before a muffle furnace burning and a MFB-TGA in DBC Chengdu laboratory analyses. Results obtained by TGA are slightly lower than results estimated by the muffle furnace. Sample in TGA being fluidized, a better mixing of air and ilmenite allow a more accurate result.

- The maximum mass gain is 3.43% at 20:00 on March 18th. It is 26.5 hours after the complete switch air/steam in FR.

- The mass gain decrease after this. This is probably due to fresh OC being injection to the system, which makes the mean $R_0\Delta x$ lower. Alternatively, the decrease in mass gain can also be due to a change in operating conditions.

4.3. Petcoke CLC results

Non Autothermal CLC for grinded petcoke injection

This test was started on 25th March with the goal of reaching CLC mode with pure petcoke (grinded) injection. The switch air/steam in FR was initiated at 14:30 on 27 March, and FR was fully subjected to steam at about 07:00 on 28 March. However, autothermal operation of the unit was not reached after the fully switch air/steam in FR. Lignite must be injected continuously in AR to try to keep stable temperature both in AR and FR. About 400 kg/h of lignite was injected in AR to keep the temperature. Two attempts by stopping lignite injection in AR have been done during this test:

- One was between 16:50 and 19:00 on 28 March 2024.
- The other was between 22:10 and 23:30 on 28 March 2024.

It means that the unit was in CLC condition during these two time periods mentioned above. Results obtained during these two periods are similar. For sake of clarity, only results obtained during the first period are presented in this section.
Main operating conditions during CLC mode with grinded petcoke fuel are given in *Table 6*.

Table 6: Main operating conditions during CLC mode with grinded petcoke as fuel

Petcoke flowrate (kg/hr)	Thermal input (MW)	OC circulation rate (t/hr)
500	5.0	116

Only steam is injected in the FR and Lignite injection in AR is used to increase the temperature in the unit before switching in CLC mode. As soon as the lignite injection in AR stopped, the temperature decreased very fast in the unit and especially in FR
This indicates that the heat brought by the OC reoxidation in AR is not enough to compensate the heat losses (in the operating conditions at this time). One major issue which can explain this phenomenon is the PSD for grinded petcoke.

Grinded Petcoke PSD analyse shows that around 74% of particles have a size lower or equal than 45 µm. For 45 µm, the theoretical efficiency of V-202 cyclone (FR cyclone) is around 60 % which is very low. It means that 40% of unburned char particles escaped from the top of the cyclone with the gas and are not recycled to the FR. This can influence considerably the reduction degree (low $R_0\Delta x$) in the FR and consequently low heat generated in AR.

To verify this hypothesis, OC has been sampled several times in the FR to evaluate the reduction degree ($R_0\Delta x$). $R_0\Delta x$ values for grinded petcoke are lower than for the other fuels injected. This clearly indicates a lower reduction degree when using petcoke grinded as fuel and could confirm the fact that part of char is lost in the cyclone gas outlet and do not contribute to the reaction in FR.

Result petcoke KPI 1

With only grinded petcoke as fuel, autothermal state has not been reached. Consequently, the temperature dropped very fast in the FR and in the entire unit. It is then difficult to find steady operating conditions since OC circulation rate and char conversion are closely linked to the temperature.

Mean operating conditions during the selected period for KPI calculation is given in Table 7 and *Figure 5*.

Table 7: Operating conditions during CLC mode with grinded petcoke as fuel during selected period.

FR temp	AR top temp	Fuel flowrate	MW	OC flowrate	CO2 AR outlet	CO2 FR outlet
924-865 °C	978-915 °C	500 kg/h	5.0	116 t/hr	≈ 7.6 %v	≈93 %v

Despite this situation, CCE calculation (see section 4.1) has been done:

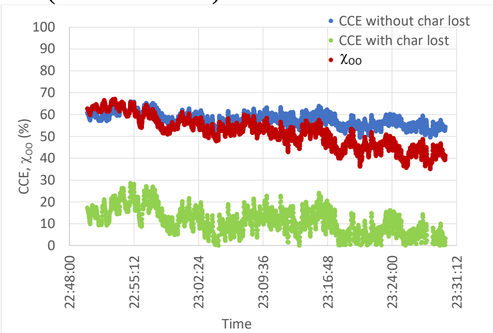


Figure 5: Evolution of CCE with time during the selected period.

It can be clearly seen on *Figure 5* that CCE is decreasing with temperature, due to lower conversion in FR.
The mean value of CCE during the selected period is 58.5 %. However, it does not make sense to calculate CCE without considering the char lost in cyclone, since this carbon will not be captured. By collecting the char lost out of the cyclone, it was possible to estimate the char flowrate evacuated with the gas out of the cyclone: around 380 kg/hr. Then, CCE has been recalculated considering the char loss: the mean value is around 12.3 %.
The low value of CCE without considering char lost can probably be attributed to the low char conversion due to char particles entrainment in the FR dense phase since grinded petcoke PSD is much lower than the design. Another hypothesis, which cannot be verified, is the soft and sticky properties of fines char particles which could be transported to the AR with OC.

Result petcoke KPI 2

Another key parameter to evaluate the global efficiency of the quality of the CLC mode is the Oxygen Demand (OD). The equations used to calculate Total Oxygen Demand KPI 2 can be found in section 4.1. The term $F_{C,elut}$ can be calculated by making a hypothesis on the char conversion in the FR. The value has been taken equal to 20 % for petcoke feedstock.
Figure 6 shows the evolution of the total oxygen demand and oxygen demand in FR over time during the selected period. It is reminded here that the unburnt gas compounds concentration at FR outlet is determined by gas sampling analysis (external laboratory in Chengdu). Consequently, the values taken are the gas sampling analysis performed during the operation and thus the variation of total oxygen demand only reflects the fuel flowrate variations during operation. The mean values under the selected period are 4.9 % for the total oxygen demand and 15.6 % for the oxygen demand in FR. Additionally, if the char lost at FR cyclone is considered, the mean values for Total oxygen demand and oxygen demand in FR reaches respectively 6.2 % and 45 %.

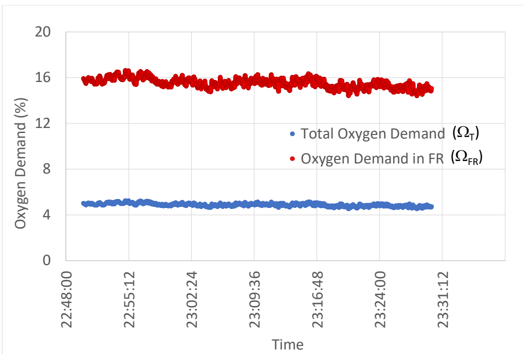


Figure 6: Evolution of OD with time during the selected period.

Since the conversion of char from grinded petcoke is probably very low, the oxygen demand in FR is then high compared with results of full lignite fuel.

Gas analyses at AR outlet

When available, O_2 and CO_2 concentration at AR outlet have been recorded through dedicated online analysis. Table 8 shows average value at AR outlet.

Table 8: Average value of gas at AR outlet on 28 March

Component	Content
CO_2	7,05 %vol
O_2	7.74 %vol

Gas analyses at FR outlet

As already mentioned previously, due to technical issues on CEMS, several gas samplings have been performed at the outlet of V-202 (fuel reactor cyclone) for further gas chromatography analysis in an external laboratory (for CO, CO_2, HC and H_2).
Table 9 shows the results of the analysis performed on a sample taken at FR outlet during CLC mode with grinded petcoke as fuel.

Table 9: Analysis Results of gas sample taken at FR outlet on 28 March at 23:40

Component	Content
CO_2	93.4 %vol
CO	2.0 %vol
H_2	3.6 %vol
CH_4	1.1 %vol

Ilmenite $R_0 \Delta x$ analysis

Like for lignite feedstock, ilmenite was sampled during petcoke feedstock operational test to measure the $R_0 \Delta x$.

OC Results from muffle furnace and TGA method

OC maximum mass gain is 1.34% at 14:00 on March 28th. The maximum of the mass gain is lower than that during the first test in which the mixture fuel lignite/petcoke was injected.
One sample collected on March 29th has been further analysed by MFB-TGA in DBC laboratory. Mass gain is quite low according to both methods. Moreover, mass gain obtained by MFB-TGA is even lower than the value estimated by the furnace. Values are 0.70% and 0.87% according to TGA and to Furnace tests respectively.

5. Conclusions

In March 2024, auto-thermal CLC mode with lignite was successfully performed, with a fired power between 3.7 and 6.5 MW. The unit temperature was stable without any supplemental fuel injection into the AR, with T_{AR} ~ 950-1050°C and T_{FR} ~ 900°C. Solid (OC) circulation in the full CLC loop was in the range 150 – 170 t/h. The oxygen flowrate was considerably above the design value of 50 t/h, being a consequence of the earlier mentioned heat losses caused by the hot spots due to refractory damages in a fuel reactor with a high dense phase level of oxygen carrier. For the same reason, also the fired power is higher than the design range of the unit, which was originally 3 – 4 MW for nominal and maximum values.
During test campaigns, some key performance indicators (KPI's) were estimated to quantify the performance of the CHEERS demonstration unit when operated in CLC mode (carbon capture efficiency and FR oxygen demand). For the lignite tests, the main results are very good:

- Carbon capture efficiency (η_{CC}): 90 – 94 %
- Oxygen demand in FR (Ω_{FR}): 2.5 – 3 %
- FR gas conversion efficiency (1 - Ω_{FR}): 97 – 97.5 %

The second test campaign consisted of in injecting 100% grinded petcoke. With this type of feedstock, autothermal CLC mode was not reached. It was difficult to maintain a stable temperature in the unit. To avoid temperature decrease, lignite was injected into the AR for supplemental firing. Petcoke injection flowrate to the FR was varying between 400 and 500 kg/h, equivalent to about 3.7 – 4.7 MW fired power. OC circulation flowrate was around 120 t/h. The evaluated KPI's were in the range:

- Carbon capture efficiency (η_{CC}): 50 – 60 %
- Oxygen demand in FR (Ω_{FR}): 15 – 16 %
- FR gas conversion efficiency (1 - Ω_{FR}): 84 – 85 %

As previously, fuel size particles impacted the performances. The grinded petcoke particle size was lower than what was used as design values. By summarizing, grinded petcoke test results could be improved by adjusting the petcoke particle size.
When compared to other CLC units in the world, the performance of the CHEERS demonstration unit is found to be very good [1]. The tests in the CHEERS demonstration unit have enabled major progress in the development of the CLC process. Many learnings on the technology have been achieved. This large-scale operation is an important step on the road to CLC industrialization.

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