

## SrCuO<sub>2</sub> THE POTENTIAL OXYGEN CARRIER MATERIAL FOR CHEMICAL LOOPING COMBUSTION APPLICATIONS

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**Abstract** - the paper shows results on chemical looping combustion (clc). Clc is one of the most promising combustion technologies among others. This is due to advantages it offers. One of them is a production of a concentrated pure carbon dioxide (co<sub>2</sub>) stream, and significant nitrogen oxides (nox) reduction emissions to the atmosphere from fuel combustion processes. Comparing to standard well known combustion methods, co<sub>2</sub> separation in clc is realized without any energy penalty for its separation. The direct contact between air and fuel is avoided, and required oxygen is delivered to the fuel with application of solid state oxygen carriers. They are typically transition metal oxides (fe<sub>2</sub>o<sub>3</sub>, mn<sub>2</sub>o<sub>3</sub>, cuo) combined with some inerts such as silica, titania etc. Which circulate between reactors: the fuel reactor (where fuel combustion reaction takes place) and the air reactor (where oc is regenerated by an air). Many studies on both synthetic and natural oxygen carriers (various ores, sediments from deep water etc.) were considered. Recently, synthetic mixed materials have been investigated intensively due to their emerging clc properties.

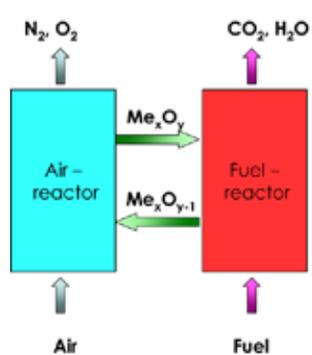
The aim of the work was to study the CLC reaction performance for a novel Cu-based oxygen carrier material (OCM). SrCuO<sub>2</sub> was prepared in a form of a fine powder using oxides and carbonates as starting materials. The preparation procedure was patented. Reactivity tests were performed under isothermal conditions for multiple redox cycles using a thermogravimetric analyzer (TG) by Netzsch STA 409 PG Lxxx. For the reduction reactions of this novel spinel type material, 3% H<sub>2</sub> was used. 3% H<sub>2</sub> was applied since it is important syngas component, obtained from gasification process of both coal and biomass. While for OCs regeneration an air was used. Both effects of the reaction temperature and redox cycles number on the reaction performance were evaluated. The examination of SrCuO<sub>2</sub> indicated an excellent and stable redox CLC behavior. Also oxygen transport capacity was investigated and it was satisfactory estimated to be 8.08 wt%. Therefore, advantage of the material was good reactivity with both fuel and air.

OCM was examined by using additional methods such as to check the regeneration ability by: Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), and Particle Size Distribution (PSD). OCM showed an outstanding recyclability and maintaining the chemical properties after carrying out the multiple redox cycles, without signs of agglomeration, and as high as 1590 °C thermal resistivity (which is attractive). The behavior of SrCuO<sub>2</sub> was compared with behavior of other well recognized OCM i.e. CuO/TiO<sub>2</sub>, which unfortunately exhibited a problematical stability.

The investigations carried out in this work supported the possible utilization of a novel Cu-based type of material such as SrCuO<sub>2</sub> for power generation.

### INTRODUCTION

Nowadays, Chemical Looping Combustion (CLC) is promising combustion method. It offers many advantages such pure stream of CO<sub>2</sub> and NO<sub>x</sub> reduction of emission from fossil fuel combustion while maintaining unchanged process efficiency (Clayton et al. 2014a). In this process direct contact between air and fuel is avoided. And oxygen is transported to the fuel reactor by oxygen carrier (OC) as shown in Fig. 1. They are typically transition metal oxides (Fe<sub>2</sub>O<sub>3</sub>, Mn<sub>2</sub>O<sub>3</sub>, CuO) combined with some inerts such as SiO<sub>2</sub>, TiO<sub>2</sub> etc. which circulate between reactors: the fuel reactor (where fuel combustion reaction takes place) and the air reactor (where OC is regenerated by an air). Many studies on both synthetic Nalbandian et al. 2011, Clayton et al. 2014b) and natural oxygen carriers (various ores, sediments from deep water etc.) (Ksepko 2015) were considered. Recently, synthetic mixed materials have been investigated intensively due to their emerging CLC properties (Wang et al. 2014).



The CLC process is realized in two reactors, namely reducer (also called fuel reactor) and oxidizer (also called air reactor). In the fuel reactor, the fuel is reacting with OC, leading to fuel oxidation to  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . Meanwhile metal oxide is reduced to the metal oxide at a lower oxidation state or to its metallic form. Afterwards water condensation, a pure stream of carbon dioxide in fuel reactor is obtained. In the next stage of a cycle, the reduced metal oxide is transported from fuel reactor to air reactor. There the reduced OC is regenerated by air (oxidation of the oxygen carrier). At the end of the redox cycle, the oxygen carrier is ready to react with the fuel in another loop.

Fig. 1 Chemical Looping Combustion.

## EXPERIMENTAL

$\text{SrCuO}_2$  was prepared by a mechanical mixing method. Primarily, mixture of  $\text{SrCO}_3$  and  $\text{CuO}$  oxides was roughly mixed with water. After that, it was heated for 20 h at  $950^\circ\text{C}$ . The grinding and calcination process was repeated to ensure the best homogeneity of material obtained (Ksepko 2015). For comparison, simple Cu-based OC was prepared as 50 wt%  $\text{CuO}$  and 50 wt%  $\text{TiO}_2$  material was prepared. Also here the mechanical mixing method was applied, with a final sample calcination at  $800^\circ\text{C}$  for 20 h.

Average shrinkage, deformation, hemisphere and flow temperatures were determined by using a high temperature oven equipped with an IR camera. The pelletized samples were placed in an oven (PR 25/1750/PIE) and heated to  $1650^\circ\text{C}$  to observe the change in the pellet shape during the heating process.

Reactivity test were carried out in a Netzsch STA 409 PG Luxx thermal analyzer (TGA), which was coupled with 403C Aëolos quadrupole mass spectrometer (QMS). The mass spectrometer was used for the analysis of the evolved gas and could detect masses between 1 and 300 amu in the SCAN or MID mode. In the TG experiments, the mass change of the metal oxide OCs was measured isothermally as a function of time. For reactivity determination 5 redox cycles were performed to determine. About 100 mg of sample was heated in an  $\text{Al}_2\text{O}_3$  crucible to the given reaction temperature. Prior experiments, both the mass of the sample and the flow of the gases were settled experimentally. This procedure was carried out to ensure that the possible mass transfer limitations during redox reactions were minimal. Once desired temperature was reached the 3%  $\text{H}_2/\text{Ar}$  was used for the reduction reaction of MeO from OC. While 20% of  $\text{O}_2/\text{N}_2$  was used for the oxidation (regeneration) reaction of the OC. Because hydrogen is one of the major synthesis gas component of that might be used for CLC for power generation, the possible utilization of a novel Cu-based type of material for this purpose was investigated in this work. Gas flow rates were 125 and 50 ml/min for reduction and oxidation, respectively with 15 min flow (Ksepko 2015). TGA was also flushed with Ar for 5 minutes before and after each redox reaction to avoid the mixing of reduction gases and air. For investigation of the temperature effect, the redox cyclic tests were carried at  $600\text{--}800^\circ\text{C}$  temperature range.

## RESULTS

Because hydrogen is one of major compounds of syngas from the biomass/coal gasification process, therefore it was utilized as a fuel in CLC tests in this work. Five cycles of reduction–oxidation TG data were obtained for the  $\text{SrCuO}_2$  oxygen carrier at the  $600\text{--}800^\circ\text{C}$  temperature range (Fig. 2). The stable performance was observed for the OC within the studied temperature range. However, some mass decrease observed at the very beginning of cycling (reaction time ca. 5 min.) may be attributed to the water content. That observation was supported by the mass spectrometer which confirmed the presence of the  $\text{H}_2\text{O}$ .

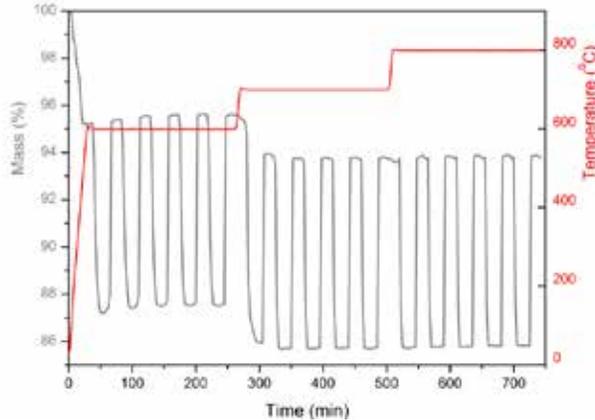


Fig. 2. 5-redox cycle TG data for SrCuO<sub>2</sub> at 600-800°C temperature range.

In Fig. 3, the five redox cycle data from TGA was shown for CuO/TiO<sub>2</sub>, which was collected at the same reactions conditions. Also here, a similar behavior during cycling start was observed. Some mass decrease was due to the presence of the water content. Nevertheless, later at temperature of 600°C the decrease in mass shows that the sample must be activated. In other words it needs at least 4 redox cycles to be completely activated. At higher temperatures (700°C), it requires at least 2 redox cycles. Based on the data from Fig. 2 and fig. 3 it may be concluded that the SrCuO<sub>2</sub> compound shows better performance than the typical simple OC with composition of CuO/TiO<sub>2</sub>. The reason for that is because SrCuO<sub>2</sub> oxide does not need any activation as it is clear based on the results presented in Fig. 2.

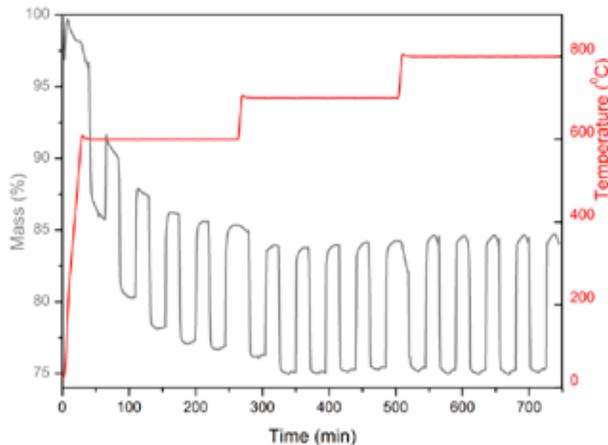


Fig. 3. 5-redox cycle TG data for CuO/TiO<sub>2</sub> at 600-800°C temperature range.

Further, one of the most important factors for selection of a solid state oxygen carrier for CLC applications was examined. That is the ability to transport oxygen to the fuel called oxygen transport capacity. The capacities were calculated for each single redox cycle for TG data recorded for the studied temperatures. The capacity stability for the SrCuO<sub>2</sub> oxygen carrier versus cycle number showed stable performance at all temperatures studied. At the lowest temperature (600°C) it showed a high capacity i.e. 7.97 wt% for the first cycle with a narrow increase up to 8.02 wt%. A similar behavior was observed for higher temperatures with final the smooth stabilization. That behavior might be due to reorganization of the pore structure during reduction-oxidation cycling because the pore structure was possibly changed with the number of cycling reactions. Further heating of the sample (up to 800°C) affected a small increase in oxygen carrying capacity of 8.08 wt%. Assuming, a negligible temperature effect on the oxygen release was

observed for the SrCuO<sub>2</sub> carrier for examined temperature range. Deducing, for SrCuO<sub>2</sub> based OCM even a reaction temperature as low as 600 °C (for gaseous fuel combustion) might be applied for low temperature CLC.

The theoretical maximum extent of mass reduction that was calculated for the SrCuO<sub>2</sub> oxygen carrier was equal to 17.47 wt% when evolving whole oxygen (that is equal to 2 moles) which is available in the chemical looping combustion reaction. For reduction of the oxide with release of 1 mole of oxygen, the theoretical extent of reduction would be equal to 8.74 wt%. For 800 °C experimental data showed capacity of 8.08 wt%. Hence, the observed mass changes might be expressed as reversible redox reactions based on the formula presented below (Eq. 1):



For the CuO/TiO<sub>2</sub> oxygen carrier, the calculated capacity stability within the cycle number differed significantly from the value observed for SrCuO<sub>2</sub>. At the lowest temperature for the first cycle, an oxygen capacity of 9.89 wt% was estimated, then the capacity decreased with an increase of the cycle number. As a result for the fifth cycle, the sample showed the lower capacity of 8.9 wt%. That value might be observed due to difficulties with releasing the oxygen and possible agglomeration of the particles. For 700 °C at the beginning of cycling, the sample showed a lower capacity (8.96 wt%) than the capacity observed at lower temperature, and the capacity also decreased with an increase in the cycle number. However, the decrease was smaller than the decrease at 600 °C. At 800 °C, an intermediate capacity was observed of 8.7 wt%. After that the oxygen capacity increased for the second cycle, and stabilized at approximately 9.6 wt% (for cycles numbered 2-5). That behavior means the temperature had some positive effect on oxygen capacity. In general, the higher the temperature, the higher the capacity that may be achieved. However, serious concerns might arise due to a possible tendency for agglomeration that may be concluded from the results shown.

As mentioned, the reactivity of the OC was evaluated based on TG data obtained from the cycling redox reactions for 600 °C, 700 °C and finally 800 °C. The calculated reduction reaction rates are shown as reaction rates at the maximum of the peak, are shown in Fig. 4. Fig. 4 shows that the reduction reaction rate increased with the increase of cycle number for SrCuO<sub>2</sub> at 600 °C, but for 700 °C, and 800 °C, the reduction reaction rate was stable. For the first reduction cycle, the estimated reduction rate was equal to 1.03 %/min, and for the fifth cycle it was 1.37 %/min. At this temperature, a continuous increase in reduction reaction rate was observed with parallel increase of cycling number. That increase of reduction rates might be as a result of the reorganization of crystal structure and pore reorganization (such as increase in size) that was supported by SEM microphotographs. The increase might possibly be the reason that the oxygen ions were easily removed from the cuprate structure.

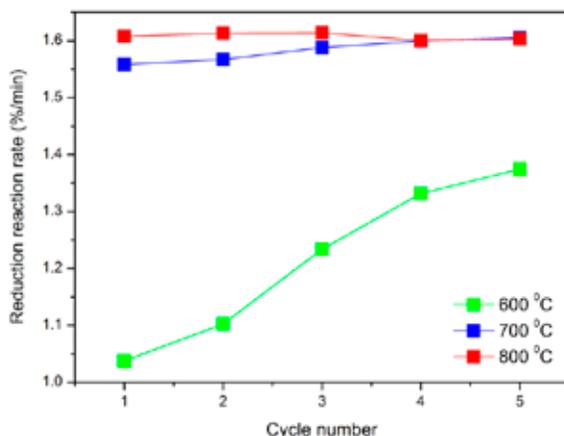


Fig. 4. Reduction reaction rate for oxygen carriers versus cycle number at given temperatures for SrCuO<sub>2</sub>.

As the temperature increased, the reduction rate also increased notable (700°C). Further heating of the sample (up to 800°C) give rise to a further increase in reduction rate. Though, the increase in rates is minor compared to the increase in rates observed between 600 and 700°C temperature. At the highest studied temperature, the maximum rate was 1.60 %/min. Therefore, observed changes of the value of the rates showed a positive effect of temperature (the higher temperature, the faster hydrogen combustion was observed). In other words, the temperature facilitated reduction of the OCM by hydrogen.

From the other hand, Fig. 5. shows reduction reaction rates calculated for monometallic CuO/TiO<sub>2</sub> OCM versus cycle number at given temperatures. The calculated rate changes do not show a convinced, and they differ much from the behavior observed for SrCuO<sub>2</sub> carrier. That is because at 600°C, the highest reduction reaction rate i.e. 3.98 %/min was observed for the first cycle, then the reduction reaction rate decreased with an increase in the cycle number up to the 3<sup>rd</sup> cycle. Then the reduction reaction rate increased and decreased again for the 5<sup>th</sup> cycle. That behavior is in contrast with the behavior observed at higher temperatures because the heating of the sample up to a temperature of 700°C leads to lower reduction rates than heating that OCM sample to 600°C. At the beginning of the cycling, 3.12 %/min rate was estimated, with an increase in rate up to the 3<sup>rd</sup> reduction cycle, and finally a significant drop-off in rates was observed. For the highest temperature (800°C), the estimated rate of 2.74 %/min was observed at the beginning of the CLC reduction reaction (1<sup>st</sup> cycle), then the intensive increase in rate, decrease and increase again for the 4<sup>th</sup> and 5<sup>th</sup> cycles and finally the stabilization of the rate at a level of 4.28 %/min (5<sup>th</sup> cycle). Therefore, in contrast to SrCuO<sub>2</sub>, for CuO/TiO<sub>2</sub>, no general rules for the effect of temperature on reaction rates was observed. The decrease in rates with cycle number might be explained by the fact that even at a temperature as low as 600°C, CuO is reduced to metallic Cu. This conclusion is based on the theoretical oxygen capacity that should have been approximately 10 wt% and agree well with the experimental data. Based on that conclusion, and because metallic Cu particles are known from literature to possess an agglomeration tendency (Clayton et al. 2014a), causing difficulty in allowing H<sub>2</sub> to penetrate through CuO particles, some reduction reaction rates were therefore observed to decrease.

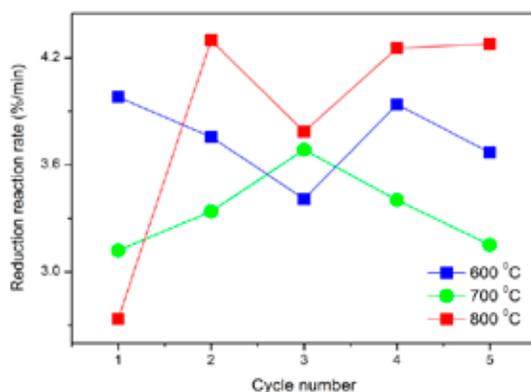


Fig. 5. Reduction reaction rate for oxygen carriers versus cycle number at given temperatures for CuO/TiO<sub>2</sub>.

Based on the calculated reduction rate values, CuO/TiO<sub>2</sub> clearly reduces faster than SrCuO<sub>2</sub> because the estimated rate ratio was equal to approximately 2. Though, the reduction stability of the simple Cu-based OCM (at examined temperature range 600–800°C) is obviously poor, making the material less predictable as an oxygen carrier material for the CLC process. For practical applications as future, potential oxygen carrier, a sufficient stability of reactivity should be provided while more suitable candidate material is selected.

For the comparison of the stability, of the newly obtained SrCuO<sub>2</sub> and simple CuO/TiO<sub>2</sub> carriers were tested under identical reaction conditions. And it is clear that standard simple CuO/TiO<sub>2</sub> showed dramatically unstable performance across the redox cycle number. As it is shown in this work, the involvement of Sr, Cu and O ions may result in a better redox CLC stability than the involvement of Ti, Cu, O obtained for the simple copper based OCM.

The oxidation reaction rates (by analogy to reduction reactions, they were calculated as a function of the cycle number) for given temperatures. They were calculated to evaluate the oxidation (regeneration) ability of the materials. A similar behavior was observed for oxidation reactions as it was observed for reduction. Because regeneration reaction rates for the SrCuO<sub>2</sub> sample increased both with a cycle number and with temperature. The oxidation rates of 1.58–1.63 %/min were estimated, and it is similar to the oxidation rates for reduction reaction. Moreover, the stabilization of rates was also observed for the fourth cycle. However, the magnitude of the effect was much smaller than the magnitude of the effect for the reduction reaction.

For CuO/TiO<sub>2</sub>, the regeneration data showed both the effect of the cycle number and temperature on sample regeneration rate. The oxidation rates were high, and they increased for cycle number 1 to 2, then the rates were stabilizing. The effect of temperature on rates was positive since 3.45, 3.77 and 4.07 %/min for 600, 700 and 800°C temperature, respectively was calculated. Overall, the oxidation reaction rates were higher about 2 times, compared to the SrCuO<sub>2</sub> rates.

## CONCLUSIONS

In this work new OC material suitable for the chemical looping processes was developed. The redox properties of the SrCuO<sub>2</sub> have been examined for CLC.

The oxygen carrier showed stable performance during the 5 redox cycle tests at temperature of 600-800°C carried out in TGA. The thermogravimetric data indicated that the developed material showed ability to react both with fuel and air. The oxygen transport capacity was determined because it is practically important parameter. At 600 °C, the determined oxygen capacity was 8.02 wt% for strontium cuprate. With observed increase of temperature a negligible increase in oxygen capacity was observed, and it has reached maximum of 8.08 wt%. Interestingly SrCuO<sub>2</sub>, as OC was able to release oxygen in temperature as low as 600°C. Moreover, reduction and regeneration reactions were fast and showed repeatable performance with CLC redox cycle number.

The advantage of the developed material is a good reactivity with H<sub>2</sub> as a fuel (a part of the syngas from the coal/biomass gasification process) that might be promising for the power generation process. The strontium cuprate material also showed a good regeneration ability with maintaining the physical-chemical properties, which was supported by SEM data. High melting temperatures determined (1590°C), and no signs of agglomeration, resulted in high temperature stability over multiple redox cycles. CuO/TiO<sub>2</sub> was investigated for comparison purposes and it has transported similar amounts of oxygen to the fuel. Though, detailed TGA study showed that cyclic stability of CuO/TiO<sub>2</sub> carrier was rather poor.

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## EXPERIENCES FROM THE OPERATION OF A FLUIDIZED-BED CHEMICAL-LOOPING-COMBUSTION SOLID-FUELS TEST RIG

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**Abstract** - The experiences with a dual fluidized-bed unit designed for chemical looping combustion of solid fuels (FB-CLC-SF) will be presented. The hot test rig of about 5 kW is constructed on the basis of the cold model with minor modifications only, which was previously investigated (Czakiert, 2016) prior to the combustion tests. The unit consists of two interconnected reactors (Fig. 1), which make up the closed loop for circulating solids (CFB). The combustion takes place in so-called Fuel Reactor (FR), which is operated under bubbling fluidized bed (BFB) conditions. The oxygen for fuel combustion is supplied by circulating solids called oxygen carriers (OC). The oxygen carriers are then regenerated in so-called Air Reactor (AR). The bottom part of AR is operated under BFB conditions, whereas in the upper part of AR a fast fluidization (FFB) takes place.

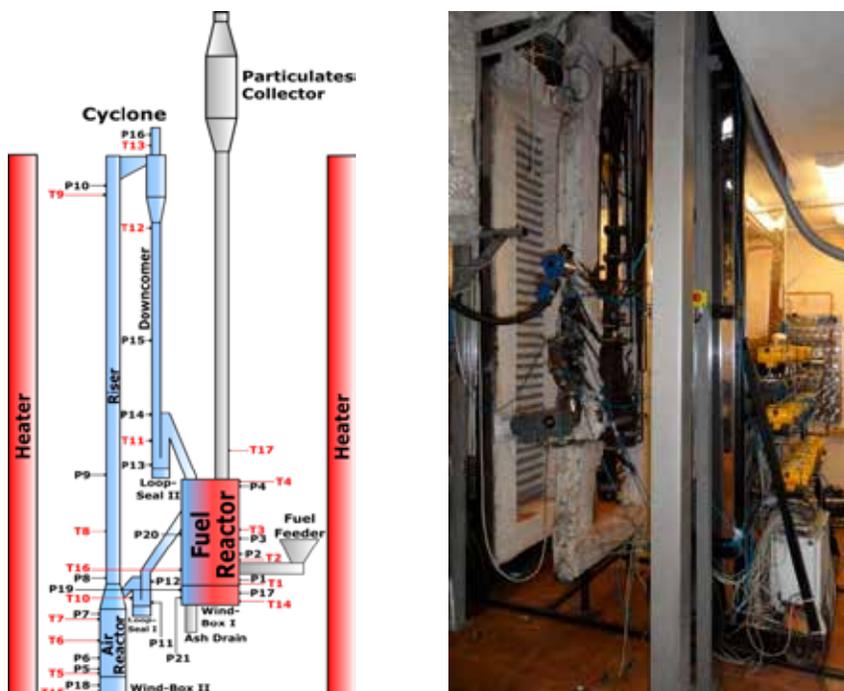


Fig. 1. The 5 kW Fluidized-Bed Chemical-Looping Combustion Solid-Fuels (FB-CLC-SF) test rig.

The solid particles in AR are fluidized with the use of atmospheric air whereas concurrently CO<sub>2</sub> is supplied to the FR and to both loop-seals. The fluidization conditions and combustion conditions are controlled and adjusted using sets of pressure sensors and thermocouples mounted on the reactors, seals and return passages (see: Fig. 1). The operating conditions are established in order to maintain smooth and interrupted fluidization as well as an efficient oxidation and reduction of OC. The composition of flue gas is determined with the use of FTIR analyzer enhanced by zirconium sensor. The oxygen concentration at the outlet of AR is measured with paramagnetic analyzer. Moreover, the solids samples are taken from both reactors and bag filter installed downstream of the FR.

Polish hard coal was used during combustion tests. The size of fuel particles ranged from 50 to 200 µm and its properties are given in Table 1. The total inventory in the whole unit is about 7 kg. The solids mass flux, which was determined experimentally, is estimated at 2 kg/(m<sup>2</sup>s).

Table.1: The properties hard coal.

M	V	A	C	S <sup>t</sup>	H	N	LHV
%	%	%	%	%	%	%	kJ/kg
13.3	29.5	8.2	61.9	1.39	3.66	0.99	23429

Three different oxygen carriers were tested under FB-CLC conditions, which are: natural CLC-type OC, Cu-based CLOU-type OC and Cu-based CLC/CLOU-type OC. The impact on the operation of dual fluidized-bed reactor as well as on the combustion performance was studied. Authors focused on the concentrations of flue gas compounds including among others: CO, C<sub>x</sub>H<sub>y</sub>, SO<sub>2</sub>, SO<sub>3</sub>, NO<sub>2</sub>, NO, N<sub>2</sub>O, NH<sub>3</sub> and HCl as well as on the quality of bottom/ fly ash, mainly in terms of the unburned carbon content. The propensity to agglomeration and sintering of oxygen carriers and fuel-ash particles were also analyzed. Concurrently, the experimental investigations were supported by the numerical simulations (Krzywanski, 2016; Zylka, 2017) performed with the 4<sup>th</sup> generation of CeSFaMB software developed by de Souza-Santos (2010), which was recently upgraded to be able to deal with the chemical looping combustion and gasification.

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