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2013

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### Recommended Citation

Marvin Kramp, Andreas Thon, Ernst-Ulrich Hartge, Stefan Heinrich, and Joachim Werther, "Simulation of the Chemical Looping Combustion Process of Coal with a Synthetic Cu-Based Oxygen Carriers" in "The 14th International Conference on Fluidization – From Fundamentals to Products", J.A.M. Kuipers, Eindhoven University of Technology R.F. Mudde, Delft University of Technology J.R. van Ommen, Delft University of Technology N.G. Deen, Eindhoven University of Technology Eds, ECI Symposium Series, (2013). [http://dc.engconfintl.org/fluidization\\_xiv/98](http://dc.engconfintl.org/fluidization_xiv/98)

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# SIMULATION OF THE CHEMICAL LOOPING COMBUSTION-PROCESS OF COAL WITH A SYNTHETIC Cu-BASED OXYGEN CARRIERS

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## ABSTRACT

A fluidized bed reactor model was implemented that allows the simulation of multiple heterogeneous and homogeneous reactions. This reactor model is used for the steady-state flow sheet simulation of the pilot plant for chemical looping combustion of the Hamburg University of Technology. The pilot plant is equipped with a two-stage fuel reactor. The simulated experiments were carried out with a Cu based oxygen carrier and German lignite dust. The developed tool is used in this study to investigate the behavior of the pilot plant and gives additional insights into the occurring phenomena. Based on the simulations it is concluded that the fuel does not mix into the bed of oxygen carrier of the lower stage of the fuel reactor which leads to large quantities of combustible gases in the off-gas of the first fuel reactor stage.

## INTRODUCTION

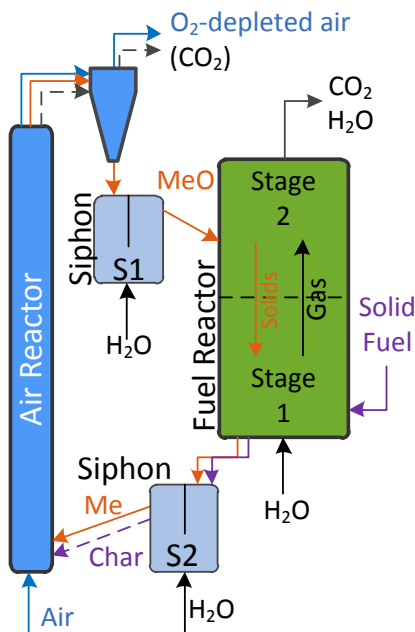


Figure 1: Scheme of the Hamburg University of Technology CLC facility.

The flow sheet simulation of solids processes is still a challenging task due to the complicated handling of distributed properties whereas the flow sheet simulation of fluid processes is state of the art. SolidSim is a steady-state flow-sheet software package for solids processes and has filled the gap in the past years. Unfortunately there is still a lack models for several unit operations like fluidized bed reactors. Hence in the course of this work a fluidized bed reactor module was developed and is applied to a complex process, the chemical looping combustion process of coal.

Chemical looping combustion allows an inherent separation of the carbon dioxide produced during the conversion of carbonaceous fuels. The process is based on a solid oxygen carrier (OC) that selectively consumes oxygen in an air reactor and provides this oxygen to the fuel conversion in the fuel reactor. A mixing of air nitrogen with the carbon containing off-gas is thus prevented.

Recently a chemical looping combustion test plant for solid fuels with a rated power of 25 kW<sub>th</sub> was erected and commissioned at the Hamburg University of

Technology. Different from most other plants a two-stage bubbling fluidized bed fuel reactor was integrated into the plant. A scheme of the plant is shown in Figure 1. The solid fuel is introduced into the lower stage of the fuel reactor and the oxidized OC coming from the air reactor is added to the upper stage. Volatiles and unconverted products of the char gasification in the lower stage have to pass through the upper stage of the fuel reactor and get a second time into contact with fresh OC particles. Hence the conversion of volatiles and the products of char gasification should be improved.

In the present investigation a Cu-based OC is used with German brown coal. First the experimental results are discussed and afterwards the simulation model is described. Finally the results of the application of the developed simulation tool are interpreted.

## **THEORY**

### **Fluid Dynamics in the Fluidized Beds**

The fuel reactor fluidized beds are divided into two zones, the bottom zone and the freeboard. The bottom zone is further divided into the suspension phase and the solids-free bubble phase. The fluid dynamics of the bottom zone are calculated according to the Werther & Wein model (1). The freeboard above the bed is treated as a single suspension phase. To calculate the elutriation from the reactors an elutriation constant approach is used (2). Assuming a relative velocity of the particles equal to the difference between the superficial gas velocity and the terminal velocity of the particles (3), the solids volume concentration above transport disengaging height is calculated. The particle size distribution is fully considered. An exponential decay of the solids volume concentration in the freeboard is assumed (4).

The air reactor is operated at high gas velocities that lead to low solids volume concentrations. The measured height-dependent pressure profile is used for the calculation of the height-dependent solids volume concentration in the air reactor. A single solid-gas suspension phase is assumed.

### **Reactions**

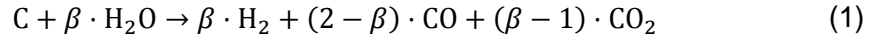
The heterogeneous reactions in the dense beds of the fuel reactors are assumed to take place solely in the suspension phase since the bubble phase is assumed to be free of solids. In the freeboards of the fuel reactors and in the whole air reactor heterogeneous reactions take place on the entire cross-section. Homogeneous gas phase reactions are allowed to take place in all gas carrying phases. The reactions lead to changes in the volume flow. For the dense beds of the fuel reactors the convective transport of gas between the suspension and bubble phase and the dilution of reactant gases by product gases is accounted for.

### **Fuel Devolatilization**

The composition of the volatiles released from the solid fuel is approximated from ultimate analyses of fuel and char according to a model by Jensen (5). The formation of nitrous oxides and the sulfur content of the fuel are neglected.

### **Char Gasification**

Char can be gasified both by  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . The net reactions are:



The factor  $\beta$  in Eq. (1) describes the ratio between the formation of  $CO_2$  and  $CO$ . Typically this value is close to unity (6) and taken as 1.2 here. Hence steam gasification leads predominantly to  $CO$  formation. The kinetic model of steam gasification is taken from Matsui et al. (6):

$$\frac{dX}{dt} = \frac{k_{1,H_2O} \cdot C_{H_2O}}{1 + k_{2,H_2O} \cdot C_{H_2O} + k_{3,H_2O} \cdot C_{H_2} + k_{4,H_2O} \cdot C_{CO}} \cdot (1 - X) \quad (3)$$

The kinetic model of char gasification by  $CO_2$  is taken from Matsui et al. (7):

$$\frac{dX}{dt} = \frac{k_{1,CO_2} \cdot C_{CO_2}}{1 + k_{2,CO_2} \cdot C_{CO_2} + k_{3,CO_2} \cdot C_{CO}} \cdot (1 - X) \quad (4)$$

$X$  denotes the char conversion and  $C_i$  are the molar concentrations of species  $i$ . Unfortunately the kinetic constants derived by Matsui et al. cannot be used since the coal used in this investigation is fine brown coal dust with a sauter diameter of 24.3  $\mu m$  while the measurements of Matsui et al. were carried out with char derived from subbituminous coal of particle sizes in the range of 297-710  $\mu m$  for steam gasification and 44-710  $\mu m$  for carbon dioxide gasification.

### Char Combustion

Char transported to the air reactor combusts with the present gaseous oxygen. Char combustion is assumed to lead solely to the formation of carbon dioxide. The kinetics are taken from Field et al. (8).

### OC Reactions

Shrinking core kinetics are applied to all OC reactions.

$$\frac{dX}{dt} = \frac{3}{\tau} \cdot (1 - X)^{\frac{2}{3}} \quad (5)$$

$\tau$  is the time until full conversion and defined as:

$$\tau = \frac{\rho_m \cdot r_g}{b \cdot k_i \cdot C_i^n} \quad (6)$$

$b$  is a stoichiometric factor.  $r_g$  denotes the grain size and  $n$  is the reaction order towards the gaseous reactant. This shrinking core approach was used to describe the reactions between the OC and combustible gases in the fuel reactor as well as the oxidation of OC in the air reactor. The reactivity of this particular OC was not investigated beforehand and hence the kinetic constants are fitted by comparison of model and experimental results.

### Cyclone Model

The cyclone separation and pressure drop is calculated according to the model by Muschelknautz et al. (9). In order to consider reactions in the cyclone solids and gases are considered well mixed, hence an ideal CSTR model is applied.

### Siphon Modeling

The steam used for fluidization of the lower siphon is distributed between the air reactor and the fuel reactor. According to previous investigations at the cold model of the experimental plant (10) it can be estimated that 80 % of the siphon gas is sent to the air reactor and 20 % to the fuel reactor. For reaction modeling the lower siphon S2 was approximated with well-mixed behavior and a solids inventory corresponding to the measured pressure drops. The steam fluidizing the upper siphon S1 does not influence the dry off-gas compositions and is thus neglected.

### Simulation Environment

The simulations have been carried out with SolidSim 1.2 (11), a steady-state flowsheet simulation environment for solids processes.

## EXPERIMENTAL

In the following sections the test plant is briefly introduced and experimental results are shown. A more detailed discussion of the experiments is found elsewhere (12).

### Plant Design

A sketch of the test plant is shown in Figure 1. It consists of a riser as air reactor and a two-stage bubbling fluidized bed as fuel reactor. The air reactor is 0.1 m in diameter and has a height of 8 m. The fuel reactor has an inner diameter of 0.25 m and each stage is 2 m high. Each stage of the fuel reactor is equipped with an overflow pipe with a height of 0.6 m above the gas distributor and an inner diameter of 0.07 m. Hence, the bed height of each stage is kept constant. Between fuel and air reactor siphons are located that separate the respective atmospheres in the reactors from each other. Each siphon is connected to a steam generator. Coal is pneumatically conveyed by a flow of CO<sub>2</sub> into the lower stage of the fuel reactor. A detailed description is given in (13).

### Evaluation

The experiments are evaluated with the help of two key performance indicators (14). The carbon capture ratio  $\eta_{cc}$  indicates the fraction of CO<sub>2</sub> produced in the air reactor instead of the fuel reactor. Char transported from the fuel reactor to the air reactor combusts to a great extent and the carbon dioxide produced is lost to the atmosphere. Hence the carbon capture ratio should be as close to unity as possible.

$$\eta_{cc} = \frac{[\dot{n}_{CO_2} + \dot{n}_{CO} + \dot{n}_{CH_4}]_{FR\ out} - [\dot{n}_{CO_2}]_{FR\ in}}{[\dot{n}_{CO_2} + \dot{n}_{CO} + \dot{n}_{CH_4}]_{FR\ out} - [\dot{n}_{CO_2}]_{FR\ in} + [\dot{n}_{CO_2}]_{AR, out}} \quad (7)$$

Another performance indicator is the oxygen demand of the gaseous products  $\Omega_{OD}$  leaving the fuel reactor (FR). It describes to what extent the products of char gasification and fuel devolatilization are oxidized in the fuel reactor by the OC particles.

$$\begin{aligned}\Omega_{OD} &= \frac{\text{O}_2\text{-demand of FR off-gas}}{\text{O}_2\text{-demand of coal}} \\ &= \frac{[0.5 \cdot \dot{n}_{\text{CO}} + 2 \cdot \dot{n}_{\text{CH}_4} + 0.5 \cdot \dot{n}_{\text{H}_2}]_{FR\ out}}{\text{O}_2\text{-demand of coal}}\end{aligned}\quad (8)$$

## Experiments

The experiments were carried out with an OC composed of 11.5 % of CuO as active ingredient impregnated on Al<sub>2</sub>O<sub>3</sub> particles (Sasol Germany, Puralox NWA155). This CuO based OC releases only negligible quantities of gaseous oxygen in the fuel reactor. A rapid solid-state reaction between the CuO and Al<sub>2</sub>O<sub>3</sub> leads to the formation of copper aluminate (15) which hinders a decomposition reaction of CuO to Cu<sub>2</sub>O. The reactivity of the OC towards combustible gases is not reduced by this solid-state reaction (15). Hence char is mainly converted by gasification and not through combustion. The fuel was a finely ground German Rhenish brown coal, provided by RWE AG. Details on fuel and oxygen carrier can be found elsewhere (12).

The siphons and the fuel reactor were fluidized with varying amounts of steam. The circulation rate between both reactors was determined indirectly from the oxidation degree of OC samples taken from both siphons (13). The off-gas was measured by continuous NDIR and heat conductivity analyzers and calibrated each day. Additionally gas samples were taken and analyzed by GC measurements. Gas sampling was done after the second stage approximately 1 m above the top end of the freeboard in the off-gas pipe. After the first FR stage gas was sampled through a port situated at the top end of the reactor but on the same axis as fuel injection. Gas was withdrawn at the reactor wall since no probe was used.

## RESULTS AND DISCUSSION

### Results of CLC Tests

An overview on the experimental conditions and results of two exemplary experiments are summarized in Table 1. In the experiments high carbon capture ratios (>96 %) and low oxygen demands (< 2.5 %) could be achieved. Another important result is that large quantities of combustible gases were measured at the top end of the first fuel reactor stage and almost fully converted in the second fuel reactor stage. The methane concentration after the first stage is low compared to H<sub>2</sub> and CO. In the experiment with the higher coal feed rate the H<sub>2</sub> concentration exceeds the CO concentration significantly while in the other experiment both concentrations are similar.

*Table 1: Experimental Results. Gas composition N<sub>2</sub>-free from GC analysis.*

<b>Parameter</b>	<b>Run 1</b>	<b>Run 2</b>
coal feed [kg/h]	5.0	3.9
CO <sub>2</sub> feed [kg/h]	3.1	3.3
feed S2[kg/h]	3.6	3.6
feed FR [kg/h]	1.7	1.3
T [°C]	900	900
$\Delta p_{AR}$ [mbar]	99	108
$\Delta p_{PC}$ [mbar]	42	43
$\Delta p_{FR}$ [mbar]	45	46
$\dot{m}_{circ}$ [kg/s]	0.136	0.145
$u_{AR}$ [m/s]	5.0	5.0
<b>FR stage 2</b>		
CH <sub>4</sub> [vol.-% dry]	0.53	0.07
CO <sub>2</sub> [vol.-% dry]	97.8	99.4
CO [vol.-% dry]	0.77	0.21
H <sub>2</sub> [vol.-% dry]	0.86	0.28
<b>FR stage 1</b>		
CH <sub>4</sub> [vol.-% dry]	3.72	1.79
CO <sub>2</sub> [vol.-% dry]	37.1	60.97
CO [vol.-% dry]	24.8	18.84
H <sub>2</sub> [vol.-% dry]	33.3	18.39
<b>Air Reactor</b>		
O <sub>2</sub> [vol.-% dry]	5.15	9.12
CO <sub>2</sub> [vol.-% dry]	0.27	0.14
$\eta_{cc}$	98.6%	99.0%
$\Omega_{OD}$	1.5%	0.34%

## Process Simulations

Since the kinetic constants for the gasification reactions and the reactions between OC and combustible gases are unknown the kinetic constants had to be fitted. The experimental run with the highest coal feed rate is used to derive the appropriate kinetic rate constants. In order to reduce the calculation time during the parameter fitting the fuel reactor was simulated independently from the air reactor and fed with a flow of oxidized OC with steady-state particle size distribution (fines free). It was possible to achieve a good agreement between the measured and simulated concentrations after the second fuel reactor stage. It was however not possible to simulate the measured high concentrations of CO and H<sub>2</sub> after the first stage (see Table 2 sim A). Hence an assessment was started with the aim to investigate the occurring phenomena.

The continuous concentration measurements were subject to fluctuations but the GC measurement generally confirmed the measured concentrations. The gas sampling after the second FR stage should be more or less free of horizontal concentration profiles due to the change of pipe diameter above the second stage. After the first FR stage gas was sampled close to the wall on the same axis as fuel injection. Hence it cannot be guaranteed that the cross-sectional average concentrations are equal to the measured concentrations at the wall. Previous studies with tracer gas injection into the upper dilute zone of a riser at the Hamburg University of Technology have shown that local differences in concentrations can be preserved over large distances (16). Hence it could be

possible that the sampled gas after the first fuel reactor stage is influenced by local effects.

The comparatively low methane concentrations are likely to be attributed to the steam reforming of methane. The following reaction is considered in the simulations in order to account for steam reforming:



This reaction is catalyzed by reduced OC as well as the reactor walls (Ni) (17,18) and could also explain the comparatively high  $\text{H}_2$  concentrations.

The used model assumes a well-mixed behavior of the solids in each stage of the fuel reactor. In case of good mixing of fuel and OC the products of gasification  $\text{CO}$  and  $\text{H}_2$  should be readily converted. Thus the high measured concentrations are possibly attributed to an isolated flow of fuel and products of devolatilization and gasification inside a plume rising through the lower stage. Cold model investigations were carried out and indeed a visible bubble flow above the coal injection port could be observed at the reactor wall. Additional simulations and calibration runs have been carried out with partial or full coal injection into the freeboard of the second stage. A calculation flow scheme for this case is shown in Figure 2. A comparison of gas compositions for different coal bypass situations is shown in Figure 3 and Table 2 (sim B/C). The simulations confirm that it is possible to raise the amount of  $\text{CO}$  and  $\text{H}_2$  in the fuel reactor off-gas significantly by assuming the coal to bypass the bed of OC in the lower stage. If all coal bypasses the dense bed the simulated  $\text{H}_2$  concentration reaches 21.4 % compared to 33.3 % which were measured. In the experiment with high coal feed rate the  $\text{H}_2$  concentration was higher than the  $\text{CO}$  concentration. This ratio was different at lower coal feed rates (see Table 1).

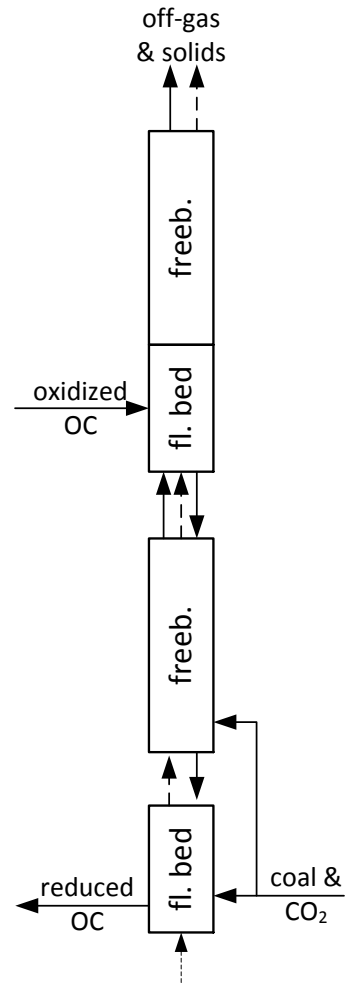


Figure 2: Schematic scheme of fuel reactor model. Dashed lines indicate gas and solid lines solids flow.



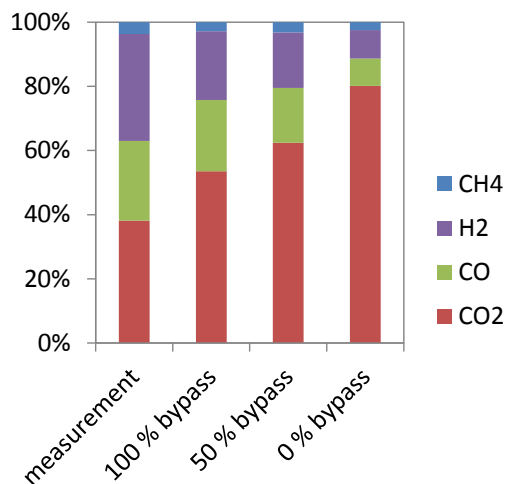


Figure 3: Simulated dry gas compositions after first FR stage for different bypass scenarios.

It was also tested whether an increased amount of fluidization steam could elevate the H<sub>2</sub> content after the first stage. The steam feed was doubled (sim D Table 2) but has only a minor influence on the H<sub>2</sub>/CO ratio. The CO content was lowered by 1.5 % and the H<sub>2</sub> concentration was increased by 0.2 %. To achieve a greater effect the ratio of steam gasification to gasification by CO<sub>2</sub> needs to be altered which was not done here. Since the OC particles present in the bottom region of the freeboard have a significant influence on the H<sub>2</sub> and CO content another study was carried out without coal bypass but also without consideration of H<sub>2</sub> and CO consumption reactions in the first stage. This measure leads to a further great increase in H<sub>2</sub> and CO concentrations after the first stage but it also leads to greater H<sub>2</sub> and CO concentrations above the second stage (3.6 % and 1.7 %, respectively). Greater amounts of char are gasified there and since virtually no OC particles are present in the top freeboard region the products of gasification cannot be converted anymore. From the experiments it is known that in fact large amounts of char are elutriated in the fuel reactor (corresponding to 7 wt.-% of the fed char). The measured concentrations were nonetheless close to zero which could either mean that the lost char is very unreactive or that in practice OC particles are present in the upper region of the freeboard. Attrition leads to a continuous production of fine particles that can be elutriated. The OC loss was determined for the considered experiments to 24.6 g/h from bag house filter samples. Assuming that 50 % of this loss originates from the fuel reactor another simulation has been set up to consider this flow of fine particles. This variation showed that the loss of fines due to attrition is too low to have a noticeable effect on the gas conversion (identical results to sim E which was the basis). Another possible explanation could be that particles ejected from collapsing bubbles at the surface of the dense bed reach higher into the freeboard than expected. Lowering the decay constant to 50 % of its original value leads to a noticeable decrease of the CO and H<sub>2</sub> concentrations after the second stage but also the concentrations after the first stage are influenced (sim F, Table 2).

Table 2: Comparison of results from experiment 1 and simulations. (A: reference case, B: 50% bypass, C:100% bypass, D: double steam feed, E:no OC reactions in stage 1, F: lowered solids decay in freeboard)

	exp 1	sim A	sim B	sim C	sim D	sim E	sim F
<b>FR stage 2</b>							
CH <sub>4</sub> [vol.-% dry]	0.5	0.3	0.6	0.4	0.3	0.1	0.1
CO <sub>2</sub> [vol.-% dry]	97.8	98.6	97.0	96.8	97.2	94.6	97.6
CO [vol.-% dry]	0.8	0.4	0.9	1.0	0.9	1.7	0.9
H <sub>2</sub> [vol.-% dry]	0.9	0.7	1.5	1.8	1.6	3.6	1.4
<b>FR stage 1</b>							
CH <sub>4</sub> [vol.-% dry]	3.7	2.4	3.2	2.8	2.5	3.4	3.8
CO <sub>2</sub> [vol.-% dry]	37.2	80.2	62.4	53.5	55.1	12.5	12.5
CO [vol.-% dry]	24.8	8.5	17.0	22.2	20.7	42.4	42.7
H <sub>2</sub> [vol.-% dry]	33.3	8.9	17.4	21.5	21.7	41.7	41.0
$\eta_{cc}$	98.6%	98.9%	98.9%	98.7%	98.8%	98.8%	99.1%
$\rho$	1.5%	1.8%	3.8%	3.3%	2.8%	4.5%	2.2%

## CONCLUSIONS

A fluidized bed reactor model was implemented that allows the simulation of multiple heterogeneous and homogeneous reactions. Hence a tool is derived that allows the simulation of a full process with a complex reaction scheme. This tool uses SolidSim as simulation framework which facilitated this investigation. The extraction of simulation results is convenient and the full support of distributed properties allowed an efficient implementation of the elutriation rate model for the prediction of the solids circulation rate between the reactors. Furthermore the implemented model for the solid separation in the cyclone could be used.

The developed tool can be used to investigate the behavior of a plant and gives additional insights into the occurring phenomena. In this study it could be shown that the lower stage of the fuel reactor operates in a different mode than expected. The mixing of fuel and OC in the lower stage is poor due to complex fluid dynamics. A different gas sampling strategy will also be necessary to get reliable information on the performance of the first fuel reactor stage. It is planned to investigate the kinetics of the occurring reactions individually in detail in order to reduce the necessary parameter fitting.

## ACKNOWLEDGEMENTS

The present work was financially supported by the German Federal Ministry of Economics and Technology (FKZ 0327844B / CLOCK) with additional funding from BASF SE, EnBW Kraftwerke AG, E.ON Energie AG, Hitachi-Power Europe GmbH, RWE Power AG and Vattenfall Europe Generation AG. The responsibility for the content of this report lies with the authors.

## SYMBOLS AND ABBREVIATIONS

$\eta_{cc}$	carbon capture ratio, - (def. by Eq. (7))	$k_{i,j}$	kinetic constants for gasification modeling, m <sup>3</sup> /s mol or m <sup>3</sup> /mol
$\Omega_{OD}$	oxygen demand of fuel reactor off-gas, - (def. by Eq. (8))	$\dot{m}_{circ}$	OC circulation rate, kg/s
$C_i$	concentration of compound i, mol/m <sup>3</sup>	$\dot{n}_i$	molar flow of compound i
		$\Delta p$	pressure drop, mbar

$u_{AR}$	AR superficial gas velocity, m/s	FR	fuel reactor
X	char conversion	OC	oxygen carrier
$\beta$	ratio of CO / CO <sub>2</sub> production in steam gasification	PC	post combustor (2 <sup>nd</sup> fuel reactor stage)
AR	air reactor		

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