Proof Of Concept Of The CaO/Ca(OH)$_2$ Reaction In A Continuous Heat-Exchanger BFB Reactor For Thermochemical Heat Storage In CSP Plants

Sylvie Rougé[1a], Yolanda A. Criado[2b], Arthur Huille[3], and J. Carlos Abanades[4]

1-Research Engineer, Commissariat à l'Energie Atomique et aux Energies Alternatives, CEA/LITEN/DTBH, 17 rue des martyrs, 38054, Grenoble Cedex 09, France

2-PhD student, Spanish Research Council CSIC-INCAR, C/ Francisco Pintado Fe, 26, 33011, Oviedo. Spain

3-Research Engineer, Commissariat à l'Energie Atomique et aux Energies Alternatives, CEA/LITEN/DTBH, 17 rue des martyrs, 38054, Grenoble Cedex 09, France

4- Chem. Eng., Ph.D., Research Professor, Spanish Research Council, CSIC-INCAR, C/ Francisco Pintado Fe, 26, 33011, Oviedo. Spain

[a] sylvie.rouge@cea.fr
[b] yolanda.ac@incar.csic.es

Abstract. The CaO/Ca(OH)$_2$ hydration/dehydration reaction has long been identified as a attractive method for storing CSP heat. However, the technology applications are still at laboratory scale (TG or small fixed beds). The objective of this work is to investigate the hydration and dehydration reactions performance in a bubbling fluidized bed (BFB) which offers a good potential with regards to heat and mass transfers and upscaling at industrial level. The reactions are first investigated in a 5.5 kW batch BFB, the main conditions are the bed temperature (400-500°C), the molar fraction of steam in the fluidizing gas (0-0.8), the fluidizing gas velocity (0.2-0.7 m/s) and the mass of lime in the batch (1.5-3.5 kg). To assist in the interpretation of the experimental results, a standard 1D bubbling reactor model is formulated and fitted to the experimental results. The results indicate that the hydration reaction is mainly controlled by the slow kinetics of the CaO material tested while significant emulsion-bubble mass-transfer resistances are identified during dehydration due to the much faster dehydration kinetics. In the continuity of these preliminary investigations, a continuous 15.5 kW BFB set-up has been designed, manufactured and started with the objective to operate the hydration and dehydration reactions in steady state during a few hours, and to investigate conditions of faster reactivity such as higher steam molar fractions (up to 1), temperatures (up to 600°C) and velocities (up to 1.5 m/s).

INTRODUCTION

Heat storage is a technical and economic key issue for Concentrated Solar Power (CSP) plants in order to match electricity production with demand at low cost. Compared to sensible or latent heat storage technologies, thermochemical heat storage (TCS) interest is based on its higher energy storage density (300-500 kWh/m^3) and its potential for longer storage periods. However the technology is far away from industrial maturity. Indeed, only a few research programs have investigated thermochemical storage at a level higher than thermogravimetric (TG) equipment. The EU FP7 Storre project (www.storre-project.eu) project was designed to cover some of these gaps of knowledge. The conceptual integration of the storage system in the solar plant and the overall energy efficiency and economic interest have also to be addressed in Storre, but are beyond the scope of this paper.

REACTION AND REACTOR

The first question to address in a thermochemical energy storage project is the choice of the chemical reaction couple. Previous reviews [1, 2, 3, 4] have identified the reactions in the range 300-600°C, most of them being solid-gas reactions. Among these reaction systems, the hydration and dehydration of natural lime was chosen as a promising reversible reaction for CSP plants because the range of temperature of this reaction, 400-600°C, was in good agreement with CSP levels of temperatures, the storage density was high, the reversibility of the reaction was proven in TGA and the products were non-hazardous, abundant and cheap. The reaction system follows:

$$CaO(s) + H_2O(v) \leftrightarrow Ca(OH)_2 (s) \qquad \Delta H^o_{298K}=-109 \ kJ/mol$$
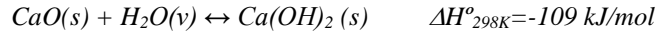
Even if these reactions have been widely studied [5, 6, 7, 8, 9], few extensive kinetics models were existing and exhibited some discrepancies [10, 11]. The reaction intrinsic kinetics was studied again [12] and revealed fast reaction rates in the minute range duration, suitable for a continuous process. As the endothermic and exothermic reactions are both gas-solid reactions and highly energetic (1850 kJ/kg of CaO), the reactor had to be designed in order to minimize the heat and mass transfer limitations and to behave as close as possible to a perfect kinetics-limited system. Moreover, the reactor had to be up scalable industrially in the 100MWth range. These considerations led to choose fluidized beds as the more promising technology, as they were widely studied in other applications [13] and are fully industrial as boilers. They can be up scaled to high velocity operations and exhibit a reasonable high thermal transfer between the bed emulsion and walls either to bring the heat during the endothermal reaction or to remove it during the exothermal one. Both circulating (CFB) and bubbling fluidized beds (BFB) are mature technologies and could be considered. Bubbling fluidized bed technology was in the end chosen because the heat transfer between the bed and a submerged heat exchanger is higher on one hand, and because it is better adapted to larger particles in the range 200 to 2000 µm on the other hand. Material properties improvement is beyond the scope of this paper but is indeed a main issue for the development of chemical storage using $CaO/Ca(OH)_2$. For example, an improved material was manufactured in the frame of the Storre project and had the shape of spheres with a diameter in the range of 500-2000 µm.

The reactor concept will thus be a bubbling fluidized bed continuously fed with the solid reactant and a fluidizing gas that transports the reactant gas (steam) coupled with a submerged heat-exchanger that will provide heat for dehydration during the charging period and recover heat of hydration during the discharging period. The conceptual design of the storage process developed in Storre is summarized on Fig.1. During the charge step, lime stored in a cold solids silo at low temperature is fed in a FB reactor where it is heated to the dehydration temperature, dehydrated and superheated, then the outcome solid is stored in a hot solids storage silo. The power input (Q_{IN}) coming from the solar field can be transported indirectly by a heat transfer fluid or directly in a particle solar receiver as suggested by some authors [14, 15, 16]. During the discharge step, the calcium oxide CaO coming from the hot solids silo at high temperature feeds the hydration FB reactor and releases both sensible heat (Q_{OUTs1} and Q_{OUTs2}) and heat of reaction (Q_{OUTr}) that are used either to heat a heat transfer fluid such as molten salts or directly to preheat, vaporize and superheat the high pressure steam of the power cycle. The outlet hydrated solid is stored again in the cold solids storage silo. This hydration heat-exchanger reactor can be arranged in a succession of perfectly mixed BFB reactors as represented on Fig.1 or have a form of a single long fluidized bed rather similar to a counterflow tube-calendar heat-exchanger [17].

A more detailed investigation of this energy storage system and its thermal integration within the solar field and steam power cycle is being investigated in the Storre project but is beyond the scope of this work. The present study is focused on the performance of the gas-solid reactor and on the hydration and dehydration reactions in the fluidized beds of Fig. 1. To our knowledge, only a previous study by P. Pardo [18] reported experimental results of lime hydration and dehydration in a 5.5 kW bubbling fluidized bed. However, the operational parameters in such study where relatively narrow when compared to realistic operating conditions at industrial scale: fine particle materials in the range 5-10 µm that could not be fluidized alone (Class C in the Geldard classification), low superficial velocity of the fluidizing gas (max 0.1 m/s), low partial pressure of steam (max 0.2 bars). The parameters targets in this work were 200-2000µm for the particle diameters, a fluidizing velocity up to 1.5 m/s, and pure steam as fluidizing gas. As the equilibrium temperature is 507°C for a steam pressure of 1 bar, the bed temperature should be above 550°C during dehydration to have a fast enough conversion rate.
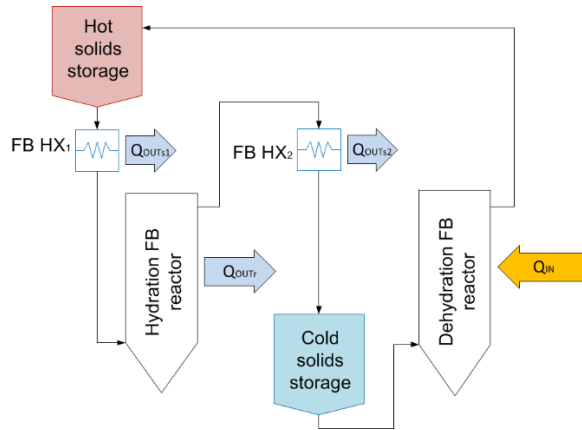
FIGURE 1. Basic schematic process view of the CaO/Ca(OH)$_2$ energy storage system.

BATCH BFB PROGRAM

Batch Experimental Setup

The objective of the Storre project was to build and operate a continuous bubbling fluidized bed with a reaction power of 5 kW. In the meantime of the design and manufacture, the facility described in [18] was upgraded to increase the gas velocity and to reduce the heat losses in the reactor. The experimental set up is based on a batch BFB, the simplified scheme is depicted on Fig.2.

The reactor bed is made of stainless steel 316L and consists of a cylinder with a diameter of 108 mm and a height of 900 mm operating slightly above atmospheric pressure. The gas distributor is made of a grid of 21 drilled screws. The fluidizing and reactive gas is a mixture of superheated dry air and steam, the partial pressure of the mixture is controlled by the upstream circuit flowmeters-regulators. At the top of the bed, a 0.55 m high conical expansion prevents excessive slug expansion and solids entrainment. A 5.5 kW and 500 mm high electric heater (heater 2 on Fig.2) placed on the external surface of the reactor provides the hydration power, the other electric heaters (heaters 1, 3, 4) compensate the heat losses. There is no cooling system, the dehydration power is dissipated by heat losses only.

The conversion of the solids during the reaction is continuously measured by the difference of the steam flowrates at the inlet and outlet of the bed. At the inlet the steam flowrate is measured in liquid phase by a mass flowmeter and at the outlet it is calculated by measuring the gas moisture and temperature thanks to 2 hygrometers H1 and H2 located respectively after the reactor and after the filters. In order to decrease the gas temperature and the residence time in the filters (90 liters), an air flowrate is added downstream the reactor. 6 type K thermocouples measure the bed temperature along its height at +40, +140, +240 and +340 mm from the distributor, and along its radius R at +0R/3, +1R/3 and +2R/3. The wall temperatures are measured by 2 thermocouples on each heater zone but cannot be considered as precise measures because of heating wires proximity. The absolute pressures are measured before and after the reactor. The electric powers of the heaters are not measured and are given by the regulator order.

The operating parameters are the initial mass of solids (1.5 to 3.5 kg), the fluidizing gas velocity (0.1 to 1 m/s), the particle size (100 to 800µm), the partial pressure of steam at the bottom of the bed (0 to 0.8 bars) and the temperature in the bed (400 to 500°C). The kinetics studies have shown that the particle diameter is not a parameter in itself, but it allows to vary the fluidizing velocity in a larger range.
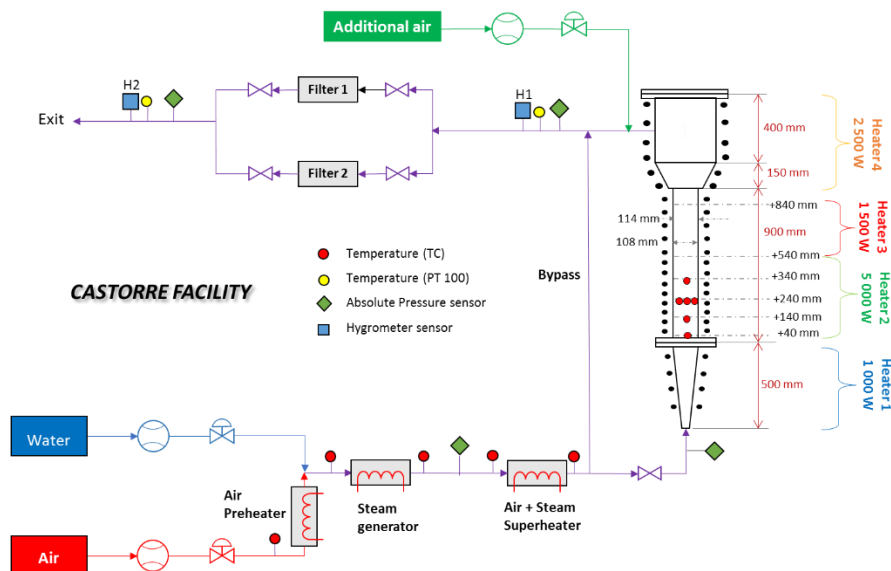
FIGURE 2. Schematic view of the batch experimental setup.

Lime Material

The lime for all the tests is a commercial grade, 95%w CaO, supplied by CARMEUSE, obtained by the calcination of $CaCO_3$ at 1000°C, separated in 4 lots of particle size by sieving, 100-200µm, 200-400µm, 400-600µm and 600-800µm and delivered in the CaO form in tight buckets.

The lime chemical activity was tested on a thermogravimetric (TG) equipment previously used for the kinetics model tests [12]. Probably due to its high calcination temperature, the lime has a moderate conversion yield of about 0.3 when calculated on the total mass and a hydration kinetics slowed by a factor of five compared to the kinetics model established on very reactive lime. The dehydration kinetics is in good agreement with the kinetics model.

Batch Experimental Procedure

In a typical test, between 1.5 to 3.5 kg of fresh calcium oxide is weighed and put in the reactor, then the reactor is closed and heated up by the reactor's heaters, this operation can last one hour due to the inertia of the system. In the meantime, the air flowrate upstream the reactor is set and heated in order to preheat the filters located after the reactor. This air flows in a circuit that by-passes the reactor in order to avoid any start of reaction in the bed of lime. When the circuits are preheated enough to avoid condensation, the steam can be started if needed. Once the system is in steady state, the bypass valve is switched off to allow the mixture of air and steam to enter the fluidized bed. This moment corresponds to the beginning of the test and the fast transient variation of the parameters. The end of the test is detected both by the transient signal of the hygrometers and the electric power of the heater 2. When the test is finished, the facility is prepared for another test by switching on the bypass valve and changing the bed temperature and steam molar fraction parameters. The same lime batch will be used for several cycles, one cycle corresponding to a full hydration-dehydration loop.

Batch Experimental Results

78 tests (reference is #n°test) were performed on the batch facility, about which 30 are completely reliable and were used for the model validation, they are summarized on Table 1. A minor number of tests have been done twice and showed a good repeatability. As some discrepancies were systematically noted between the signal of the hygrometers and the inlet water flowrate when no reaction was occurring, even after calibrating the hygrometers, a procedure of in-situ calibration was applied: the steam flowrate calculated from the hygrometers was corrected by a constant value for each test in order to have a good agreement during the bypass mode or after the end of the test (i.e. when the reaction has ceased). A typical dehydration test (#45) and hydration test (#52) are shown on Fig.3. For

simplicity, the parameters that do not vary (fluidizing velocity, air flowrates and electric power of heaters 1, 3 and 4) are not shown. The inlet and outlet steam flowrates, the bed temperature and the electric power of heater 2 are given versus time.
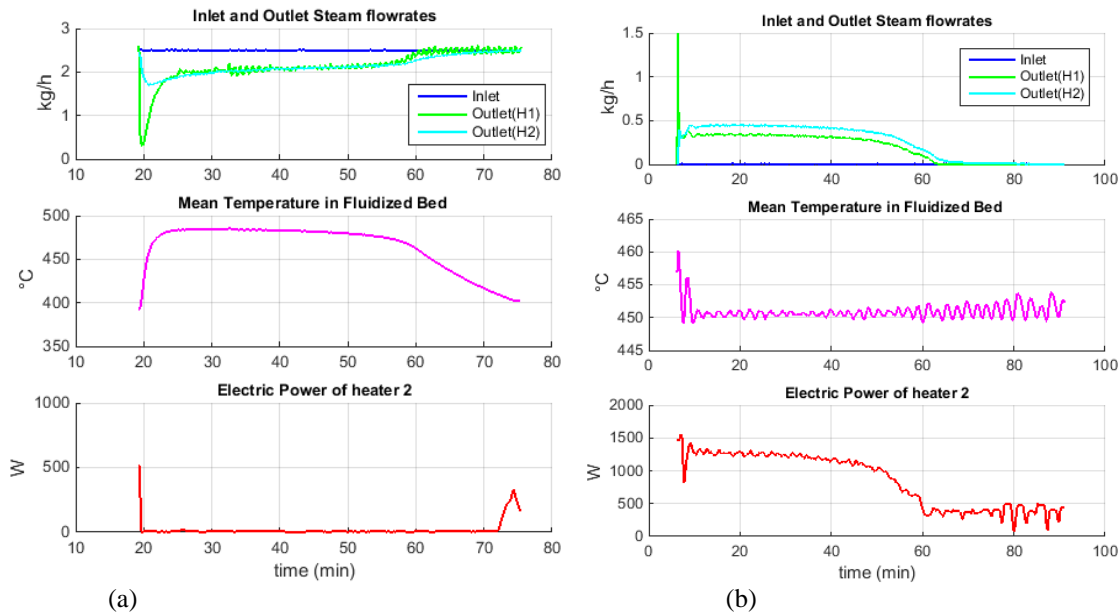

FIGURE 3. Evolution of the main test parameters during hydration #45 (a) and dehydration #52 (b)

The hydration reaction starts at +20 min when the mixture of steam and air start to fluidize the bed, the hygrometers show a sharp reduction of the steam flowrate downstream the reactor indicating that steam is consumed in the reaction. As the bed has no cooling sources, the bed temperature increases from 400 to 480°C and the electric heater compensates this internal source of heating by switching off the reactor heater. The increase of the bed temperature slows down the reaction speed as the operating conditions are closer now to the reaction equilibrium curve than at the beginning of the test. At +60 min, the steam depletion and the bed temperature are decreasing, showing that the reaction will soon be complete, it ends at +65 min.

The dehydration conditions are more stable as the bed temperature is controlled by the electric heater power. The dehydration starts at +8 min when the air flow enters and fluidizes the bed. The dehydration has probably started during the heating of the fixed bed to the dehydration temperature in the bypass mode, as indicated by the sharp signal of the hygrometer H1 at the beginning of the test. Then the two hygrometers measure a steady production of steam, indicating that the dehydration takes place. The bed temperature is stable at 450°C because the dehydration heat is provided by the increase of the electric heater. At +62 min, the production of steam stops and the electric heater power decreases, indicating that the reaction is complete.

Parameters Influence On The Reaction Activity

When the parameters of the reaction are stable during the test, their effect can be highlighted by comparing directly the tests results. As explained earlier, it is easier to compare the dehydration tests because the bed temperature is steady. The main parameters are the bed temperature, the inlet partial pressure of steam, the fluidizing velocity and the mass of lime in the reactor. The particle diameter is not considered as a parameter on its own as it showed no effect on the reaction speed in TG tests, but it allows to increase the fluidizing velocity. In the test matrix of Table 1, dehydration part, the temperature influence can be evidenced by comparing tests #52 and #55, or #63, #50 and #58 or #44, #60 and #48. The steam pressure effect can be evidenced by comparing tests #63 and #44 or #50 and #60 or #58 and #48.The fluidizing velocity effect can be evidenced by comparing tests #52 and #63 or #55 and #58. The mass of active lime can be evidenced by comparing tests #52 and #73 or #63 and #78. The direct comparison depicted on Fig.4 shows that the dehydration is enhanced by a high temperature, low steam fraction, low mass of solid and high fluidizing velocity and that the effect is higher when the reaction is slow.

HYDRATION tests								
Particle size (µm)	200-400				600-800		200-400	
Mass of Batch (kg)	2.15				2.00		3.5	
Fluid. Velocity (m/s)	0.4		0.2		0.7		0.4	
Molar fraction of steam	0.5	0.8	0.5	0.8	0.3	0.5	0.5	0.8
T bed (°C) initial 400	# 45 # 62	# 61	# 57		# 70	# 68	# 74	
430			# 54					
450	# 43	# 40		# 65				# 76
480	# 51	# 49 # 67						

DEHYDRATION tests								
Particle size (µm)	200-400				600-800		200-400	
Mass of Batch (kg)	2.15				2.00		3.5	
Fluid. Velocity (m/s)	0.2		0.4		0.7		0.2	0.4
Molar fraction of steam (-)	0	0.08	0	0.08	0	0.05	0	
T bed (°C) 450	# 52		#63 # 46	# 44			# 73	# 78
480			# 50	# 60				
500	# 55		# 58	# 48				
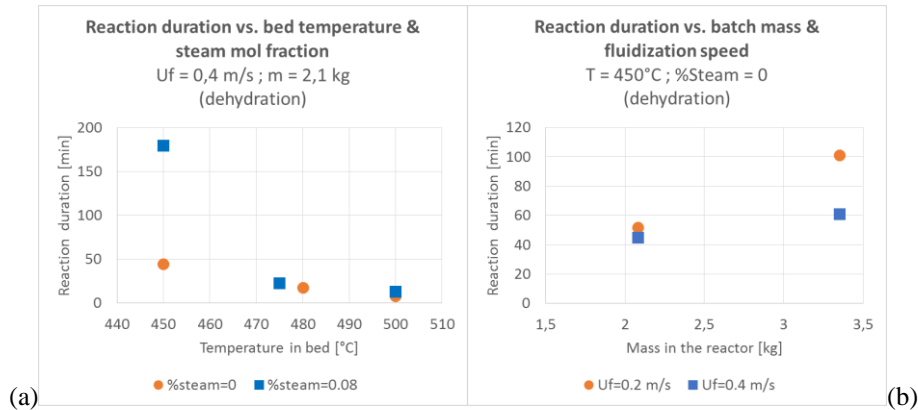
FIGURE 4. Influence of bed temperature and steam fraction (a), mass of active lime and fluidizing velocity (b) on the dehydration reactivity

Comparison To The Reactor Model

The reactor model developed in this work for the hydration of CaO and the dehydration of $Ca(OH)_2$ is a classic bubbling bed model described elsewhere [19]. The model calculates an axial profile of partial pressure of water vapor in the bubbling phase (free of solids) as a result of the gas-solid reactions taking place in the emulsion phase (assumed to be minimum fluidization conditions). A certain exchange of gas is allowed between the bubble and emulsion phase. The main variables and equations of the model are described in [19].

Figure 5 show experimental curves representing the molar $H_2O_{(v)}$ flow measured at the outlet of the reactor (dots) and the predictions given by the model (lines) described above for some selected tests of hydration and dehydration shown in Table 1. In all cases, a single cross flow factor of 1.5 has been applied to fit the experimental results shown

in Fig.5. As can be seen, there is a good agreement between the experimental results even with this simplified model when using this single parameter. The low X_{factor} value required compared to others reported for other reactor systems (typically around 3 [20]) indicates a relatively poor gas exchange efficiency between the bubble and emulsion phases during the tests performed in this experimental facility.
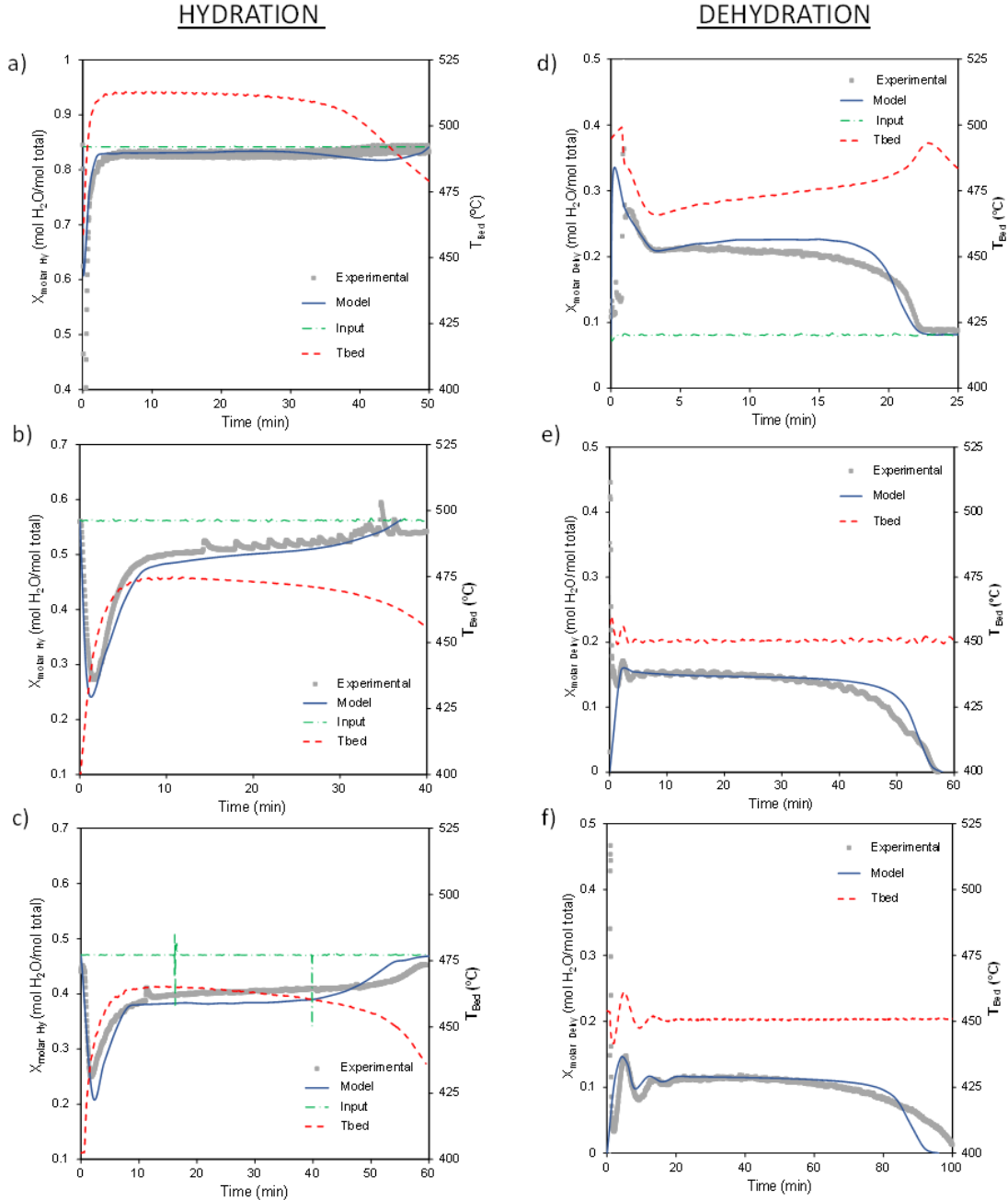
FIGURE 5. Experimental and model-predicted molar fractions of $H_2O_{(v)}$ (X_{molar}) at the outlet during hydration (a-c) and dehydration (d-f) vs time for different experimental batch tests (a #21, b #57, c #74, d #60, e #52 and f #73). In all cases the X_{factor}=1.5. The input molar fraction of $H_2O_{(v)}$ and bed temperature (T_{Bed}) are represented as dotted lines for reference.

In summary, the CaO/Ca(OH)$_2$ hydration/dehydration reaction for future energy storage systems has been successfully investigated in a batch fluidized bed operated at high gas fluidization velocities (0.2-0.7 m/s),

temperatures of around 400-500ºC and $H_2O_{(v)}$ partial pressures of up to 0.84. Valuable information has been obtained for validating a standard bubbling reactor model by using just one single parameter, the cross flow factor, to model the experimental results obtained. The batch facility presents also some design limitations, for instance to operate tests under pure steam and to control the bed temperature during hydration, leading to some difficulties in analyzing the results. Therefore, a second facility was designed with the main objective to operate in a continuous and steady-state mode during a few hours

THE CONTINUOUS BFB PROGRAM

Continuous Experimental Set Up

The continuous bubbling fluidized bed facility aims at studying the chemical reactor of lime hydration and dehydration in steady-state conditions and for a range of parameters larger than the batch facility. In particular, the dehydration can be done under pure steam, and particles as big as 1.5 mm can be fluidized. The facility is designed for a reaction power of 5 kW, corresponding to a maximal flowrate of solid of 20 kg/h, it can be operated during 3 hours at the maximal flowrate. The facility was designed for a small one-cell reactor. The technological solutions to move the solid might not be the best one at industrial scale, especially the diluted pneumatic transport that drives the solid from the reactor to the filter is indeed not the best economic choice. Many other solutions could be preferred at large scale, such as mechanical transport or dense phase transport, which is not feasible for a reactor with a volume of 7 liters and a solid flowrate of 20 kg/h.

FIGURE 6. Schematic view of the continuous experimental setup.

The reactor is similar to the batch BFB reactor and is a cylinder 108 mm of diameter and 780 mm of height, with no disengagement zone, it is made of Inconel 600 alloy to sustain temperatures as high as 800°C on the walls and is designed for a maximal pressure of 1.5 bars. It is heated by 2 main electric heaters of 6.5 kW each (Heaters 2 and 3) and a smaller one of 2.5 kW (Heater 4) and several other smaller heaters that compensate heat losses and preheat the inlet solid, the total heating power on the reactor is 20 kW. The reactor bed is cooled down by a submerged heat-exchanger designed to remove 4 kW in the worse conditions, the heat transfer fluid (max 60 Nm^3/h) is cold or warm air. The fluidizing gas is either pure air (max 40 Nm^3/h) or pure steam (max 32 kg/h) or a mixture of these two gases. The air is preheated up to 200°C in a heater of 2.5 kW, the steam is vaporized and superheated up to 250°C in a steam generator of 28 kW, the mixture of fluidizing gas is superheated to the bed temperature in a superheater of 3.5 kW (Heater 1) just below the entrance of the bed. The gas is introduced in the reactor through a heated cone

(Heater 1, 2.5 kW) and a gas distributor formed by 21 drilled screws. The air and steam circuits allow to set the fluidizing velocity and the steam molar fraction at the inlet of the bed.

The solid powder is continuously and steadily fed during several hours by a feeding system including a hopper of 80 liters, a cold regulating feeding screw, a rotary valve and a warm transport screw connected to the lower part of the reactor. A tight rotary valve prevents the hot steam and air to flow from the reactor to the hopper. In the hopper, a screw prevents the arching risks. The mixture of solid and gas is extracted from the reactor by a diluted pneumatic line cooled by natural convection and connected to a high temperature filter (max 200°C) where the gas is separated from the solid. To have a constant transport velocity close to 20 m/s, hot air (max 90 Nm^3/h) is added to the mixture of gas and solid at the outlet of the reactor, this addition of air helps to cool down the gas-solid flow before the filter and to avoid condensation when the reactor is operated under pure steam. The solid is continuously removed from the filter volume by a system including a rotary valve and a hopper. The tight rotary valve prevents the hot air and steam to flow from the filter to the hopper. After the filter, 2 hygrometers measure the gas moisture and allow, by difference with the inlet steam, to calculate the instantaneous reaction conversion yield. The solid flowrate is measured by the rotation speed of the regulating screw after calibration tests on one hand and by the weight sensors set on each hopper on the other hand.

Three thermocouples type K at 4 levels measure the bed temperature, 2 thermocouples type K measure the external wall temperature near each heater, the pressure is measured before the gas distributor, at 4 levels in the bed and after the filter. The electric power of all the heaters is measured. The cooling power of the heat-exchanger is calculated from the air flowrate and the inlet and outlet air temperatures. A sampling line allows to sample solid from the reactor during a test and to control the conversion yield by weighing the sample before and after full dehydration in a laboratory oven.

FIGURE 7. Photo of the insulated facility (left) and of the BFB reactor (right)

Lime Material

The lime provided for the continuous tests is a commercial grade, 95%w CaO, supplied by CARMEUSE, obtained by the calcination of $CaCO_3$ at 1000°C and sieved to separate the fine and big particles, the particle size range is 200-800 µm. The chemical behavior of this lime is similar to the lime used during the batch tests. 1000 kg of calcium oxide CaO were delivered in 5 tight barrels of 200 kg each.

Continuous Experimental Procedures

The air flowrates are set to their test values and the facility is preheated using the air flows and the wall heaters until the temperature on the hygrometers is close to 100°C, then the steam generator is started if steam is used during the test. The heat-exchanger circuit is switched on for hydration tests only. When the temperatures, flows and velocities are steady at their set value, the test can begin and the solid is fed to the reactor by switching on the feeding motors and setting the rotating speed of the regulation screw to the desired value. No bypass mode is necessary in this continuous procedure. The hopper n°1 starts to empty. When the screws and the reactor are full, the transport of solid starts and the hopper n°2 starts to fill. When the hopper n°1 is empty, the test is complete. The

solid is transferred from the hopper n°2 to the hopper n°1 by gravity, the 2 hoppers being the one above the other, and a new test can be prepared.

Continuous Experimental Results

The commissioning of the continuous facility was done in May and June 2016 with no major difficulty. The solid was transported successfully in cold and in hot conditions at several flowrates from 5 to 20 kg/h. The air and steam circuits were tested up to their design flowrates and temperatures. In July, one dehydration test was done under pure air with a solid flowrate of 12 kg/h, a bed temperature of 550°C and a gas velocity of 0.6 m/s. Two hydrations were done for the same solid flowrate and gas velocity at 400°C, respectively with a steam fraction of 0.5 and 1 (pure steam). The first experimental results should be available soon.

Conclusion

Following the batch experimental and model program, a continuous BFB facility was successfully designed, manufactured and started in the frame of the Storre European project (www.Storre-project.eu). This facility should provide more relevant experimental studies in order to validate a thermochemical BFB reactor model and to scale-up bubbling fluidized bed reactors for thermochemical energy storage applications based on the $CaO/Ca(OH)_2$ reversible reaction loop.

ACKNOWLEDGMENT

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