



Investigation of the applicability of Shrinking Core Model on a pilot-scale Steam Hydrator Reactor

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Abstract : Calcium looping is one of the most promising technology for Carbon Capture and Storage (CCS) purposes. It based on the reversible reaction between sorbent Calcium Oxide (CaO) and Carbon dioxide (CO₂). One of the major limitations of this process is that the sorbent loses its activity over multiple cycles. In order to improve the activity of sorbent, intermediate steam hydration step was proposed. This process is based on another reversible reaction where deactivated sorbent is treated with steam to form Ca(OH)₂. This Ca(OH)₂ undergoes the backward reaction to give back the regenerated sorbent. Several studies have shown encouraging results of the efficiency of this process. Recently, Wang et al. (2013)¹ successfully designed a fluidized bed reactor on a pilot-scale. Blamey et al. (2016)² developed a shrinking core model based on studies carried out on a small experimental reactor. In this study, we are extending the application of this model on a pilot-scale reactor. Modelling of this unit is essential for scale-up and optimization purposes. The model successfully predicts the trends when the operational parameters like steam partial pressure and operating temperature were changed.

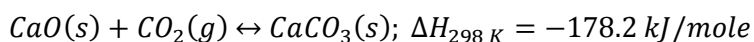
Keywords : CO₂ capture, Calcium Looping, Steam Hydration, Shrinking core model.

Introduction

Over the last few centuries, anthropogenic activities are leading to unusual phenomenon, which is degrading the environment. Global Warming is one of the most serious threats posed to our environment due to the huge increase in emissions of greenhouse gases like carbon dioxide. A recent study by International Energy Agency (IEA) showed that coal-based power plants are a major source of CO₂ emissions³. As a result, government regulations are enforced on such plants to limit the emission of CO₂. The industries employ Carbon Capture and Storage (CCS) systems to adhere to the set regulations.

The most widely used technology to carry this out is Amine scrubbing. It is popular because it can be easily retro-fitted with the existing power-plants to include carbon capture in its process. One of the major disadvantages of this process is the energy penalty associated with it. To overcome this problem many alternative technologies have been proposed namely: Membrane separation, Calcium Looping, Chemical Looping, Cryogenic Technology, etc. None of these technologies have been implemented on an industrial scale.⁴

Among all of the technologies mentioned above, Calcium looping seems to be one of the most promising. Shimizu et al. (1999)⁵ proposed this novel process of removing CO₂ from flue gas using a twin fluidized bed reactor. It makes use of the reversible reaction shown below:



A schematic showing the Calcium looping process is shown below:

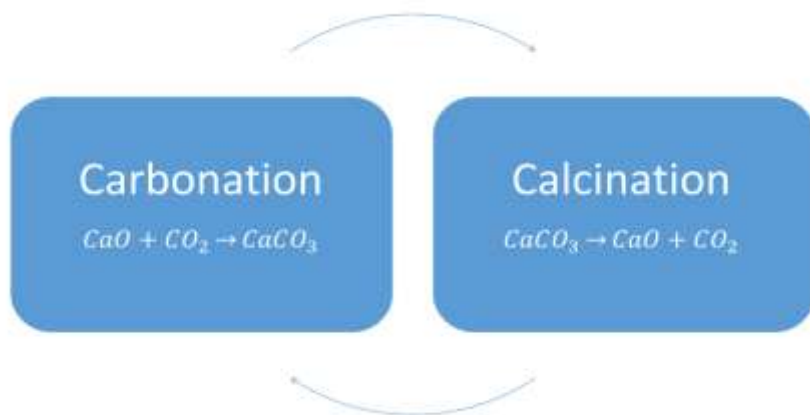


Fig. 1: Schematic of Calcium looping process

The twin fluidized bed reactors are called carbonator and calciner. In the carbonator, the flue gas is passed through a fluidized bed of calcium oxide (sorber) at 873 K (600°C), where the CO₂ in the flue gas reacts with CaO to produce calcium carbonate which is then sent to the calciner. The CO₂ lean flue gas exits the carbonator. Then the calciner is supplied with heat by providing coal and pure oxygen. It favors the backward reaction and CaCO₃ decomposes to regenerate the sorber and also releases CO₂ rich flue gas. This is then sent for further processing and sequestration.

This is a cyclic process which would regenerate the sorber CaO at the end of the calcination step. Later it was found that the sorber tends to lose its activity after a few cycles.⁵This can be attributed to the following reasons:

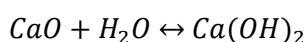
- (a) Thermal sintering of the sorber, as it was subjected to very high temperatures.
- (b) Attrition while carrying out the Fluidized Bed Operations.

This has a major impact on the Carbon capture efficiency and cost of the operation. Thus, focus of much of the research in this field shifted to preserving of sorber activity. The research activity in this regard can be broadly classified into two broad categories:

- (a) Enhancing the activity of CaO sorber by treatment methods.
- (b) Use of alternative sorbers.

Two major sorber treatment methods have been proposed. They are Recarbonation and Steam Hydration. This makes the Calcium looping process a three-stage one. Out of these two, extensive studies have been carried out on steam hydration as it was a promising option.

Steam Hydration of the sorber was first proposed by Manovic et al. (2008)⁶ to improve its activity. It is based on the reversible reaction given below:



A schematic showing the 3-stage Calcium looping process involving steam hydration is as follows:

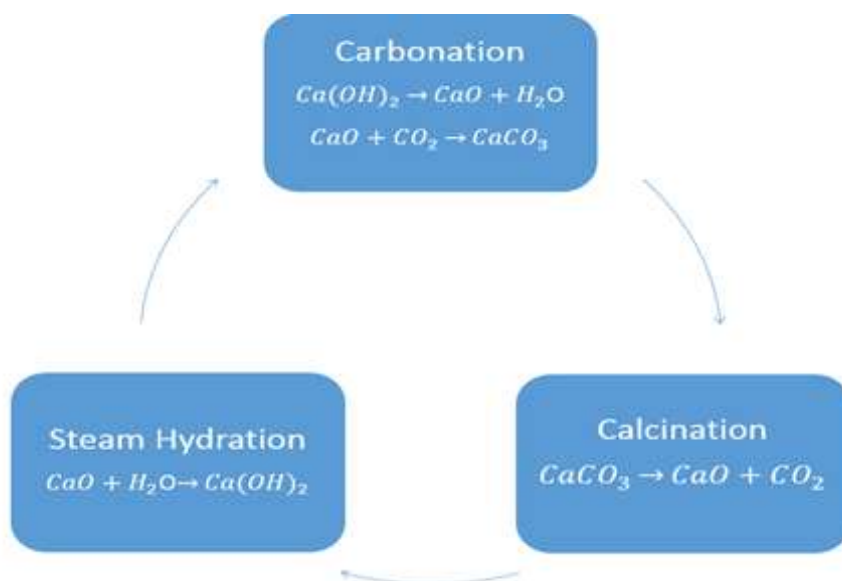


Fig. 2: Schematic showing 3-stage Calcium looping process.

They carried out experiments in a 75kW calcium looping pilot plant. Steam hydration was carried out at 100°C and atmospheric pressure. The hydrated samples were investigated using Thermo-Gravimetric (TGA) and X-Ray Diffraction (XRD) analysis. The pore surface area, pore volume distribution and swelling showed promising results for hydration. However, one of the major disadvantages observed in the sample was that it was more prone to attrition. Thus, an optimal trade-off between hydration and carbonation is highly desirable.

Ramkumar and Fan (2010)⁷ carried out a thermodynamic and experimental analysis of 3-stage Calcium looping process with steam hydration. They concluded that operation of the steam hydrator reactor can be carried out below 500°C at atmospheric pressure. For steam partial pressure of 4 atm, hydration occurs at temperatures of 600°C. This enables us to produce high-quality heat which can be used to generate extra electricity. Although, effect on sorbent because of thermal sintering at such high temperatures needs to be investigated. On carrying out steam hydration at 600°C and total pressure ranging from 8-21 atm, they reported that carbon capture capacity of the sorbent increased from 20% to 45% by weight. Phalak et al. (2012)⁸ then investigated the activity of sorbent when subjected to high-temperature steam hydration in a fixed bed reactor. They demonstrated that there was a negligible reduction in carbon capture capacity over multiple cycles. Wang et al. (2013)¹ then designed a bench-scale fluidized bed reactor to carry out steam hydration for sorbent reactivation. Cold-flow tests were carried out and reaction parameters were examined. Internals were installed to promote fluidization of fine Geldart C (CaO) particles which are difficult to fluidize. Satisfactory fluidization was obtained with the aid of rotating agitator and vertical baffles. This led to a significant improvement in hydration conversion. A linear correlation was observed between hydration conversion and sorbent reactivity. They also discussed the potential for process heat integration to be applied to this process for a variety of applications.

Then Blamey et al. (2016)² established a shrinking core model for steam hydration process describing the kinetics of the process. They reported that it fits the experimental data well. In this work, we are extending the model so that it can be applied to a pilot-scale fluidized bed reactor.

Mathematical Model

2.1 Assumptions

An extension of the shrinking core model proposed by Blamey et al. (2016)² has been used here. The main assumptions of the model are listed below:

1. The reaction occurs at the interface of unreacted core of CaO and the porous product layer of Ca(OH)₂
2. Non-equimolar counter diffusion occurs from bulk phase to the particle surface

3. Overall particle size remains constant throughout the reaction ie the product layer is non-flaking.
4. Solid particles are treated as isolated spheres.
5. Reaction kinetics are assumed to be first order.
6. Reaction is the controlling step.
7. The temperature is assumed to remain constant through the reaction.
8. Steam is an ideal gas.

A schematic representing the shrinking core model is as follows:

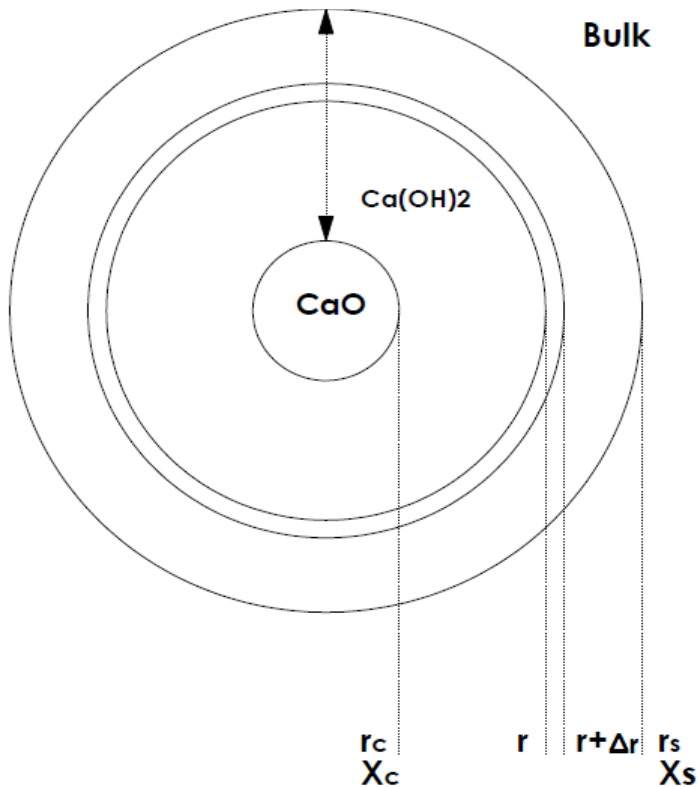


Fig. 3: A schematic representing shrinking core model.

2.2 Calculation of Mass Transfer co-efficient

The gas-phase diffusivity was calculated using Wilke-Lee correlation. The correlation is :

$$D_{AB} = \frac{10^{-4}(1.084 - 0.249 \sqrt{\frac{1}{M_A} + \frac{1}{M_B}}) T^{1.5} \sqrt{\frac{1}{M_A} + \frac{1}{M_B}}}{p t_{AB}^2 f(\frac{kT}{\epsilon_{AB}})} \quad (1)$$

Air-steam mixture was used in the experiments carried out by Wang et al. (2013)¹. The data for molecular separation at collision, energy of molecular attraction and collision function was provided by Treybal⁹. Using the obtained values of diffusivity co-efficient, the value of Schmidt number was estimated. The fluid properties of steam were obtained from Heat and Mass Transfer Data Book¹⁰. The table below provides the estimated values of diffusivity co-efficient and Schmidt number.

Table 1: Calculated values of diffusivity and Schmidt Number (Sc)

Temperature (K)\ Properties	Density of steam (kg/m ³)	Viscosity of steam *10 ⁻⁶ (Ns/m ²)	D _{AB} (m ² /s)*10 ⁻⁵	Schmidt Number (Sc)
473	0.464	15.89	5.44	0.63
573	0.384	20.01	7.81	0.67
673	0.326	24.32	1.03	0.72

Then, Sherwood Number was estimated using Frössling correlation: $Sh = 2 + 0.6Re^{0.5}Sc^{0.33}$. (2)¹¹ Then, mass transfer co-efficient was obtained using the dimensionless relation for Sherwood number, $k_g = \frac{D_{AB} Sh}{d_p}$. (3) The table below provides the details of the experimental conditions as reported by Wang et al. (2013)¹ and the estimated mass transfer co-efficient for each experiment.

Table 2: Overview of experimental conditions and calculated values of mass transfer coefficient for each experiment

Expt. no	Solid feed (mol)	S:Ca	P _{H2O} (atm)	Temp. (K)	Steam feed (l/min)	u _o (cm/s)	Re	Sh	k _g
1	14	1.28	1.0	573	28.3	3.72	0.0143	2.06	8.04
2	14	2.56	1.0	573	56.6	7.45	0.0286	2.09	8.16
3	14	3.83	1.0	573	85.0	11.18	0.0429	2.11	8.24
4	14	2.04	0.8	573	45.3	5.96	0.0228	2.08	8.12
5	14	1.53	0.6	573	34.0	4.47	0.0172	2.07	8.08
6	14	3.12	1.0	473	56.6	7.45	0.0435	2.11	5.74
7	14	2.17	1.0	673	56.6	7.45	0.029	2.08	10.71

This mass transfer co-efficient can be used to estimate the flux of steam at the surface of the particle by using the following relation: $J_{H_2O,surface} = -k_g C(x_B - x_S)$ (4)

2.3 Estimation of Effective Diffusivity

The mechanism of diffusion through the product layer was established by Blamey et al. (2016)². It was reported that Knudsen diffusion also takes place within the product layer. The data for Knudsen diffusion was taken as reported by Blamey et al. (2016)². The effective diffusivity was estimated taking into account both Fickian diffusion and Knudsen diffusion using the standard expression for resistances in series.

$$D_e = \frac{\epsilon_{Ca(OH)_2}}{\tau_{pore}} \left(\frac{1}{D_K} + \frac{1}{D_{AB}} \right)^{-1} \quad (5)$$

Here, the value of pore tortuosity to consider the non-linear nature of the pores was taken to be 3. This is a typical value suggested by Cussler¹², was to be taken in absence of experimental data.

Table 3: Calculated values of Effective Diffusivity

Cycle No.	Ca(OH) ₂ porosity	Knudsen 473 K	Diffusivity 573 K	673 K	Effective 473 K	Diffusivity 573 K	673 K
0	0.372	3.45 x10 ⁻⁶	3.80 x10 ⁻⁶	4.11 x10 ⁻⁶	4.02 x10 ⁻⁷	4.49 x10 ⁻⁷	4.90 x10 ⁻⁷
2	0.196	2.79 x10 ⁻⁶	3.08 x10 ⁻⁶	3.33 x10 ⁻⁶	1.73 x10 ⁻⁷	1.94 x10 ⁻⁷	2.11 x10 ⁻⁷
6	0.134	2.13 x10 ⁻⁶	2.34 x10 ⁻⁶	2.54 x10 ⁻⁶	9.16 x10 ⁻⁸	1.02 x10 ⁻⁷	1.11 x10 ⁻⁷
13	0.104	1.66 x10 ⁻⁶	1.83 x10 ⁻⁶	1.98 x10 ⁻⁶	5.58 x10 ⁻⁸	6.21 x10 ⁻⁸	6.73 x10 ⁻⁸

2.4 Model equations and implementation

For a shrinking core model, the general conversion equation for a spherical particle is as follows¹³,

$$X_{Ca(OH)_2} = 1 - \left(\frac{r_c}{r_s} \right)^3. \quad (6)$$

Blamey et al. (2016)² carried out a mass balance in the product layer to derive an expression of flux of steam in the product layer. The boundary conditions are: {1} At, $r = r_s x_{H_2O,r} = x_s$ and {2} At, $r = r_c x_{H_2O,r} = x_c$. The expression for flux was arrived at in the following form:

$$J_{H_2O,r} = \frac{-CD_e \ln\left[\frac{1-x_s}{1-x_c}\right]}{r^2\left(\frac{1}{r_s} - \frac{1}{r_c}\right)} \quad (7)$$

Substituting, $r = r_s$ and $r = r_c$ in equation (7), we get the flux at the particle surface (8) and unreacted core respectively (9).

$$J_{H_2O, r_s} = \frac{-CD_e \ln\left[\frac{1-x_s}{1-x_c}\right]}{r_s\left(1 - \frac{1}{r_c/r_s}\right)} \quad (8)$$

$$J_{H_2O, r_c} = \frac{-CD_e \ln\left[\frac{1-x_s}{1-x_c}\right]}{r_s\left(\left(\frac{r_c}{r_s}\right)^2 - \frac{r_c}{r_s}\right)} \quad (9)$$

From equations (8) and (9), a relation between the flux at the surface of the particle and at the unreacted core was obtained.

$$J_{H_2O, r_s} = J_{H_2O, r_c} \left(\frac{r_c}{r_s}\right)^2 \quad (10)$$

Blamey et al. (2016) then carried out a mass balance on Ca(OH)_2 formation during reaction. This gives a relation between conversion of Ca(OH)_2 and flux of steam at the unreacted core.

$$\frac{dX_{\text{Ca(OH)}_2}}{dt} = -\frac{3}{r_s} (1 - X_{\text{Ca(OH)}_2})^{2/3} \frac{M_{\text{CaO}}}{\rho_{\text{CaO}}(1 - \epsilon_{\text{CaO}})} W_{H_2O, r_c} \quad (11)$$

Since, a first order reaction is taking place and it is related to flux of steam at the unreacted core, the following equation was arrived at:

$$J_{H_2O, r_c} = -\frac{r_c S_V k_A C}{3} (x_C - x_E) \quad (12)$$

Here, S_V is the BET surface area per unit volume which can be calculated from the expression presented in equation (13)

$$S_V = S_{\text{BET}} \rho_{\text{CaO}} (1 - \epsilon_{\text{CaO}}) \quad (13)$$

Substituting, equations (4) and (12) in equation (10), we obtain a relation between the mole fraction of steam at the surface of the particle and the unreacted core which is presented by equation (14)

$$x_s = x_B - \frac{r_c S_V k_A}{3k_g} (x_C - x_E) \left(\frac{r_c}{r_s}\right)^2 \quad (14)$$

Equations (6), (9), (11), (12) and (14) are required to completely solve the model and obtain simulation results. The unknowns in the system are x_s , x_c , r_c and $X_{\text{Ca(OH)}_2}$. A script was written in Matlab to solve this system of equations. Equation (11) is an Ordinary Differential Equation (ODE) which was solved using Euler's method. Using this initial condition, the ratio of $\frac{r_c}{r_s}$ can be estimated using equation (6). This obtained value of ratio was then used to solve equations (9), (12) and (14) to obtain the values of x_s , x_c and J_{H_2O, r_c} . The obtained value of J_{H_2O, r_c} was then used to solve the ODE to give us the value of conversion for the next time interval. This algorithm was repeated to obtain a time-series data of conversion.

The parameters required to solve this system are: C , D_e , r_s , S_V , k_A , k_g , x_E and x_B . Concentration C of steam was estimated using ideal gas equation as partial pressure of steam is known. The value of k_A was taken from Blamey et al. (2016)² and equilibrium mole fraction can be calculated from equilibrium partial pressure. Equilibrium partial pressure was estimated using the relation provided by Schaube et al. (2012)¹⁴.

$$\ln\left(\frac{p_{eq}}{10^5}\right) = -\frac{12845}{T_{eq}} + 16.508 \quad (15)$$

In the experiments carried out by Blamey et al. (2016)², calcination was carried out for 15 minutes in a calcium looping cycle. However, in the experiments carried out by Wang et al. (2013)¹ calcination was carried

out for 2 hours. It has been reported in the literature that intensity of calcination affects the sorbent properties. The time of calcination is one of the parameters that affects the intensity of calcination. As a result, no. of cycles for our study was approximated to 8 because 15 minutes of calcination carried by Blamey *et al.* (2016)² is 1/8th of 2 hours of calcination carried out by Wang *et al.* (2013)¹. Since, Blamey *et al.* (2016)² provided data for only 0, 2, 6 and 13 cycles, the parameters for number of cycles being 8 were estimated using linear interpolation.

A table summarizing all the input parameters is given below.

Table 4: Summary of parameters used for modelling

Property	Value
Mean particle Diameter (μm)	20
Cross-section area of reactor (cm^2)	126.67
Sorbent Density (kg/m^3)	3350
BET Surface Area (for N=8) (m^2/g)	4.508
CaO porosity (for N=8)	0.562
Effective Diffusivity (for N=8) (m^2/s)	7.73×10^{-8} (473 K); 8.6×10^{-8} (573 K); 9.35×10^{-8} (673 K)
Rate constant per unit area (for N=8) (m/s)	3.01×10^{-7} (473 K); 1.28×10^{-7} (573 K); 8.73×10^{-8} (673 K)

Results and Discussions

4.1 Simulation Results

Plots of projected conversion versus time for the seven experiments carried out by Wang *et al.* (2013)¹ are shown below.

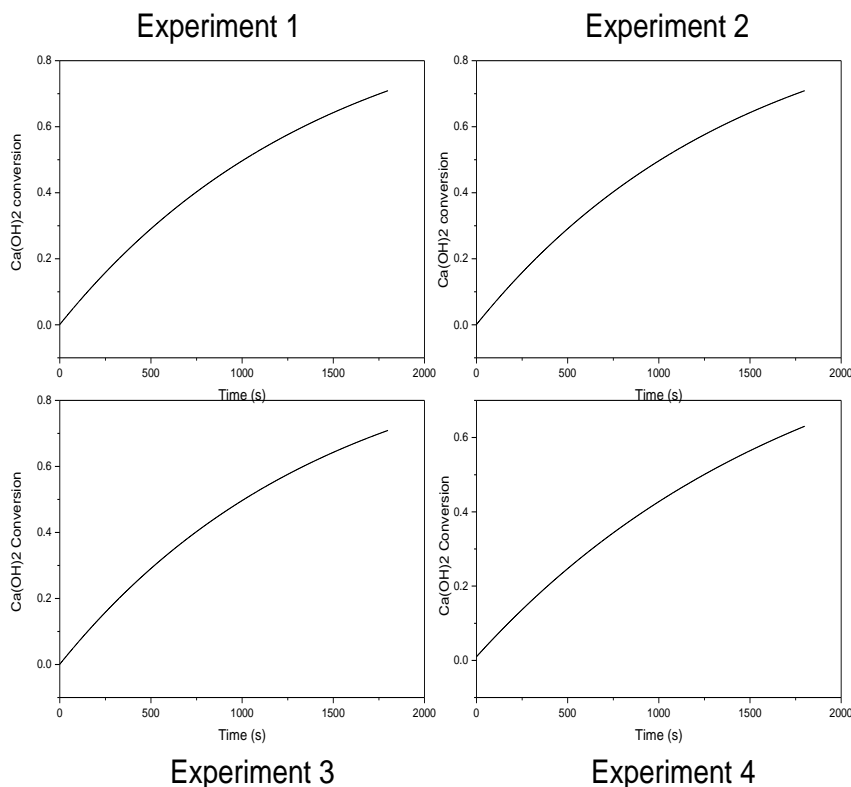


Fig. 4(a): Plots of estimated conversion as a function of time as calculated by the model developed for actual experiments 1-4 as carried out by Wang *et al.* (2013)

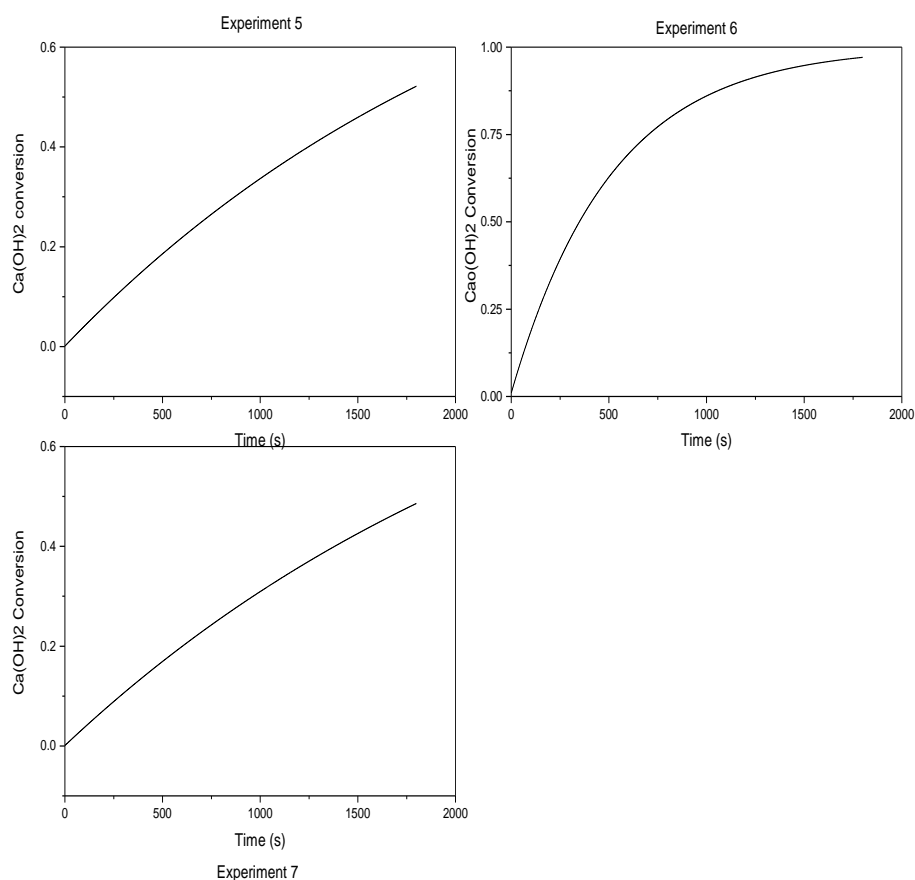


Fig. 4(b): Plots of estimated conversion as a function of time as calculated by the model developed for actual experiments 5-7 as carried out by Wang et al. (2013)

4.2 Validation with experimental data.

Wang et al. (2013)¹ carried out a detailed study of the reactor performance and effect of various parameters on the reactor performance. The parameters that were investigated are listed below:

1. Effect of Steam Partial Pressure
2. Effect of Operating Temperature
3. Effect of Steam flow-rate

In this sub-section, we have made an attempt to validate these experimental findings with our simulation results.

4.2.1 Reactor Performance study

As listed in the literature review section, the sorbent calcium oxide belongs to the family of Geldart C particles. Geldart C particles are most difficult to fluidize. As a result, baffles, agitators and other internals are required to aid fluidization. The simulation does not take into account the difficulty in fluidizing CaO particles. From Figure 5 it can be observed when time is close to zero, the model under-predicts conversion value. This can be attributed to the use of Euler's method as it is not accurate enough. At higher time, the model over-predicts the conversion value. This can be attributed to the fact that the difficulty in fluidizing sorbent has not been taken into account. Another reason can be the fact that the model does not take into consideration the pore blockage that might occur after certain extent of reaction. However, the model captures the trend of conversion with time reasonably well.

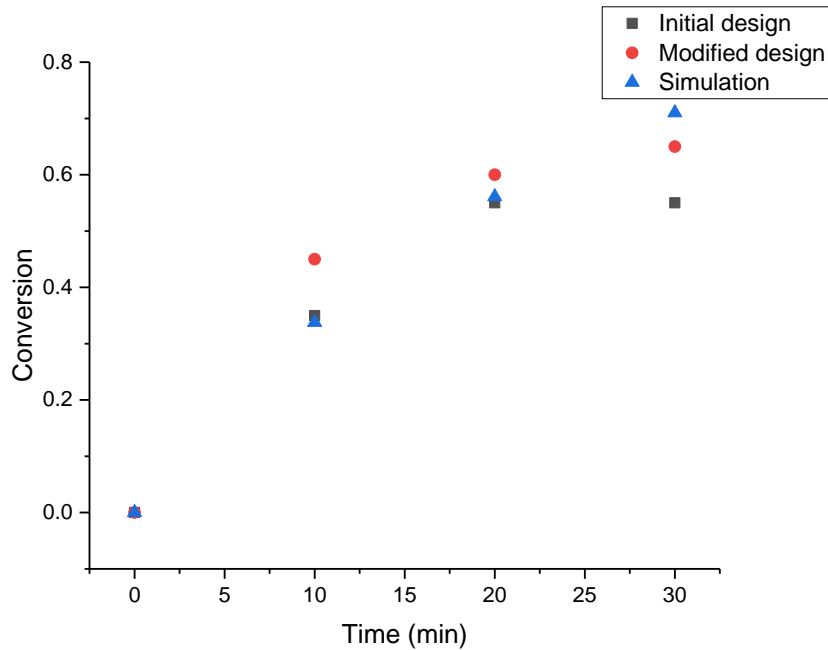


Fig. 5: Conversion versus time with respect to reactor design

4.2.2 Effect of Steam partial pressure

While carrying out experiments, it was reported that conversion is directly proportional to the steam partial pressure used to carry out the reaction. From Figure 6, it can be observed that the model successfully captures the effect of change in steam partial pressure on conversion. Although, as stated in the previous section, the model slightly over-predicts the conversion value at higher times.

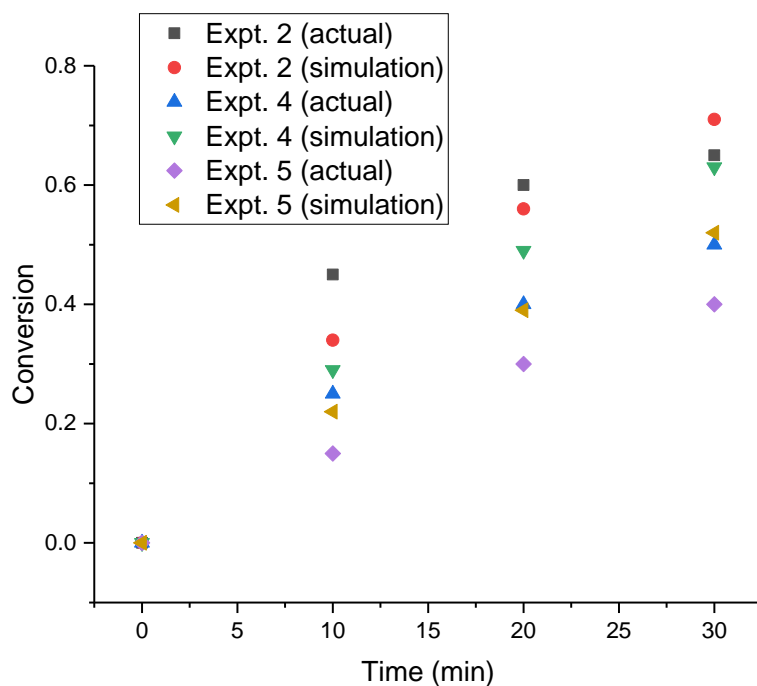


Fig. 6: Conversion versus time with respect to steam partial pressure

4.2.3 Effect of Operating Temperature

It has been observed that this reaction undergoes Anti-Arrhenius behavior. The conversion decreases on increasing the operating temperature. Again in Figure 7, a similar trend was observed between simulated and actual results. The model over-predicts the conversion at higher times. In this case also, the model successfully predicts the lowering of conversion with respect to temperature.

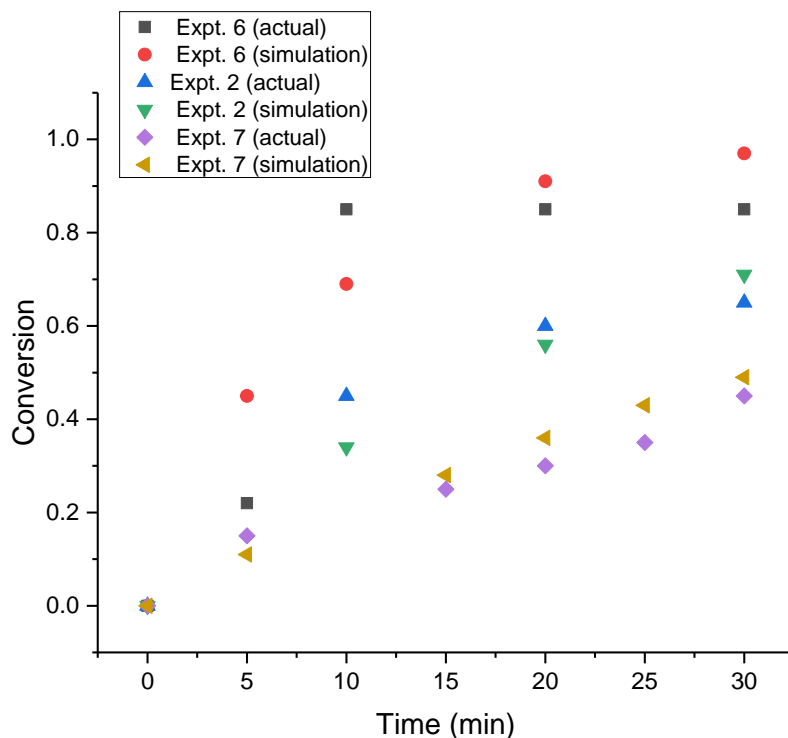


Fig. 7: Conversion versus time with respect to temperature.

4.2.4 Effect of steam-flow rate

As evident from Figure 4(a), the conversion versus time profiles for the first three experiments, where the steam-flow rate has been varied are strikingly similar. As reported in Wang et al. (2013)¹ the final hydration conversion lies within 5 percentage points of each other and more investigation of this parameter is warranted. As a result, we decided not to pursue comparison studies in our simulation for this parameter.

4.3 Limitations of the model

The limitations of this model are listed below:

1. A more rigorous hydrodynamic model taking into consideration
2. A more accurate method than Euler's method's use needs to be investigated
3. CaO particles are fine in nature. Some amount of elutriation might take place which needs to be studied further.
4. This operation is non-isothermal and its effect needs to be taken into consideration in the model
5. The particle swells up during hydration but in our model overall particle size was assumed to be constant.
6. Pore blockage that might occur during reaction has not been taken into consideration.

Conclusion & Future Work

A fluidized bed reactor model has been extended successfully to a pilot-scale reactor. The experimental trends reported by Wang et al. (2013)¹ have been successfully validated. The model successfully captures the trends of change in conversion when two of the major operating parameters are changed: (1) Steam partial

pressure and (2) Temperature. Although, the model does not predict conversion at higher times accurately. The reasons for the same have already been discussed in the previous section. In future, we hope to incorporate a more rigorous hydrodynamic model which considers the difficulty in fluidizing Geldart C particles like CaO and the elutriation of fine particles that might occur in the reactor.

Modelling of this unit is essential as it might aid us in future keeping process scale-up in mind. It can also help us in reducing the number of experiments being carried out to carry out optimization studies. Temperature is a very important operating parameter, keeping in mind the costs involved and the flow of process in Calcium looping operation. Thus, temperature optimization studies are of great importance and can be carried out using this model to estimate the optimum temperature at which steam hydration must be carried out in order to obtain the maximum conversion at lowest possible costs.

Nomenclature

C	Molar concentration	mol/m ³
D _e	Effective diffusivity within pores	m ² /s
D _{AB}	Gas-phase diffusivity	m ² /s
D _K	Knudsen diffusivity	m ² /s
d _p	Particle diameter	m
d _{pore}	Pore diameter	m
k _A	First order rate constant for the reaction of CaO with steam	m/s
k	Boltzmann constant	J/K
k _g	Mass transfer coefficient	m/s
M	Molar mass	g/mol
N	Number of cycles	
n	Number of moles	mol
P	Pressure	Pa
R	Universal gas constant	J/mol/K
r _{AB}	Molecular separation at collision	nm
Re	Reynolds number	
r	Radius	m
S _V	Specific, BET, surface area	m ² /m ³
Sc	Schmidt number	
Sh	Sherwood number	
T	Temperature	K
t	Time	s
u _o	Fluid velocity	m/s
W	Molar flux	mol/m ² /s
x	Mole fraction	
X _{Ca(OH)₂}	Conversion to Ca(OH) ₂	
ε _x	Porosity of species	
λ	Mean free path	m
μ _g	Fluid viscosity	kg/m/s
ρ _g	Gas density	kg/m ³
ρ _x	Density of species	kg/m ³
τ _{pore}	Pore tortuosity	
ε _{AB}	Energy of molecular attraction	J
$f\left(\frac{kT}{\epsilon_{AB}}\right)$	Collision function	
Subscripts for C, r, X		
c/C	At the core of the particle – CaO/Ca(OH) ₂ interface	
s/S	At the surface of particles	
B	In the bulk phase (not applicable to r)	
E	At equilibrium (not applicable to r)	

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