In-situ visualization of structural transformation during thermochemical reaction between CaO and water vapor

Rakesh Sharma*, Marie Gollsch, Christian Brack, Marc Linder **

German Aerospace Centre (DLR), Institute of Engineering Thermodynamics, Stuttgart, Germany // * rakesh.sharma@dlr.de; ** marc.linder@dlr.de

Background and Motivation

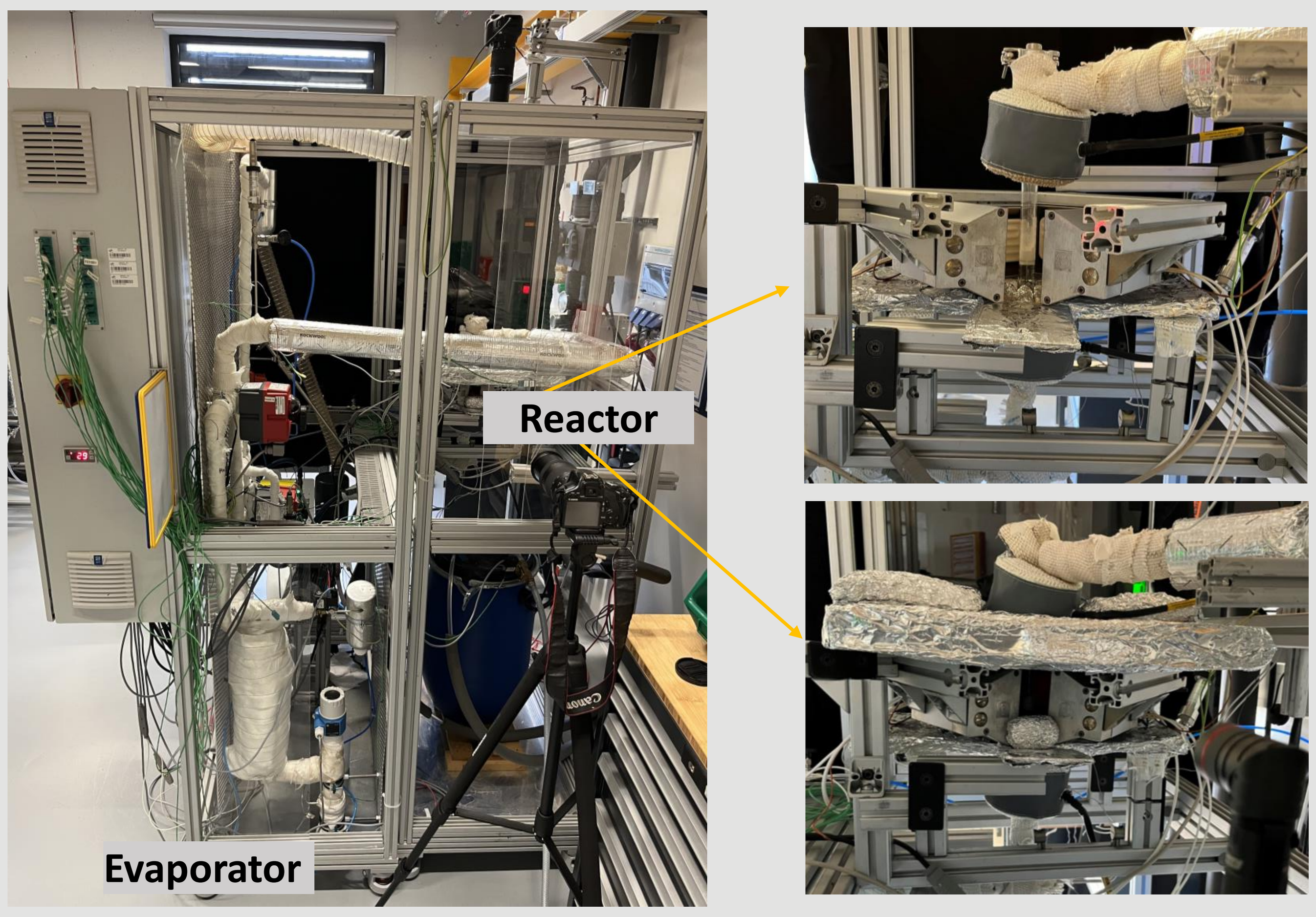
- Thermochemical de-/hydration of $Ca(OH)_2$/CaO promise an appealing method for thermal energy storage due to high energy storage density, longer storage period, minimum loss, easy availability and no danger to the environment.

$$\rightarrow \quad \Delta H + Ca(OH)_2 \rightarrow CaO + H_2O \quad / \quad CaO + H_2O \rightarrow Ca(OH)_2 + \Delta H \quad \rightarrow$$

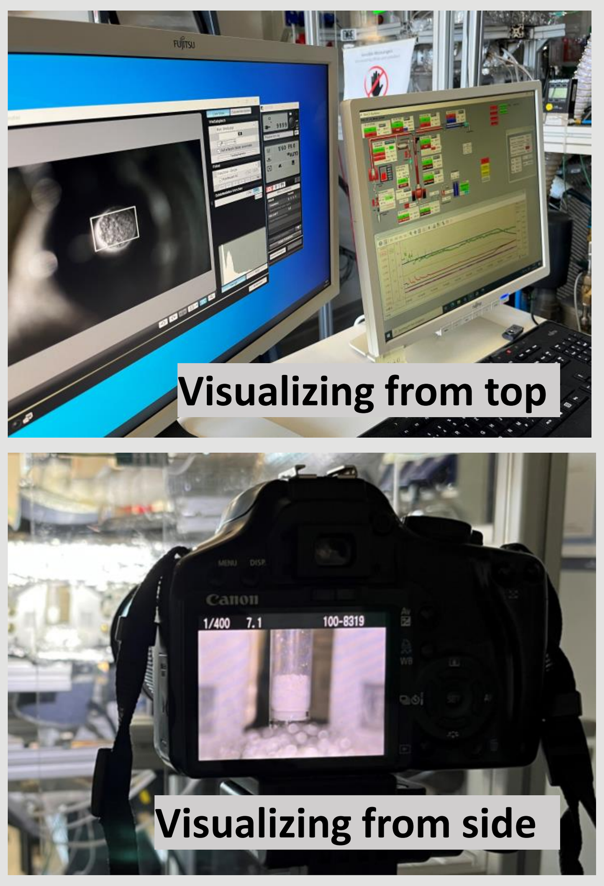
- Agglomeration and channeling are commonly observed during the reaction [1-3]
- Formation of agglomerates affects the homogeneity of reactive solid, channeling causes free flow passage for reactive gas
- Both factors tend to influence heat and mass transfer in the reactive solid and consequently the conversion and reaction rate

Approach

- An in-situ visualization approach is adopted to understand agglomeration and channeling during thermochemical reaction of $Ca(OH)_2$
- The study is carried out during several cycles of de-/hydration where starting and end state is $Ca(OH)_2$
- For visualization of sample, quartz glass-tube (withstand up to ~1300 °C and ~3 bar gauge pressure) is used. Infrared heaters are used to maintain the required temperature. Sapphire viewport is fixed at the top to visualize the top surface
- The experiments are conducted on powder bulk samples of ~2 g



Overview of the experimental setup



Experimental setup and procedure

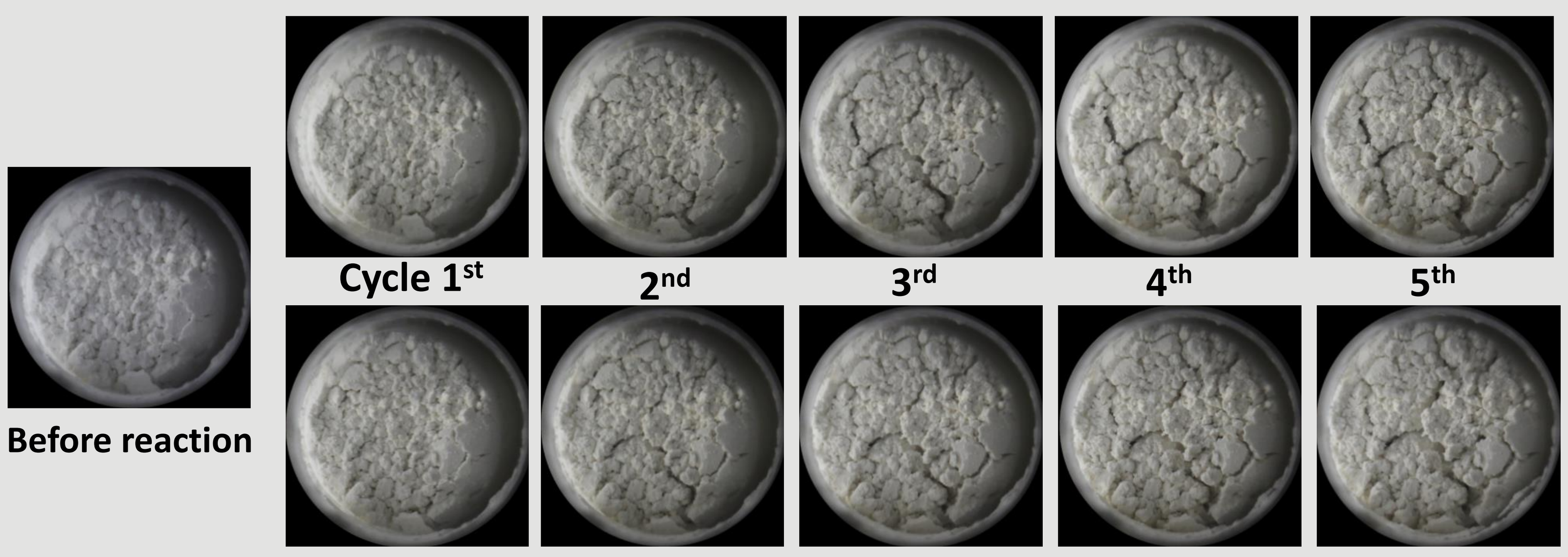
- The sample of $Ca(OH)_2$ is filled in the quartz glass-tube to visualize its structural changes
- Infrared heaters surround the reactor with a narrow opening on one side intended for visualization
- Dehydration is carried out at ~500 °C (or more) where pressure difference is created at the downstream of sample using vacuum pump to remove the dehydrated vapor
- During hydration, water vapor is supplied from the top where its flow rate is controlled by limiting the downstream pressure using vacuum pump
- Two digital cameras are used, from the top as well as side, to capture the pictures/videos of the sample material

Results and conclusions

- In-situ visualization of thermochemical de-/hydration of $Ca(OH)_2$/CaO to understand the structural changes during distinct process
- Cracks are observed at the top surface after few initial cycles which are attributed to the expansion and collision of sample particles during hydration, then followed by contraction during dehydration

Before reaction

After 10 cycles



Pictures after de-/hydration in successive cycles; top: dehydration, bottom: hydration

- Restructuring taking place continuously with prolonged cyclic reaction resulting in further cracks in other locations of the sample
- This visual investigation of structural transformation can also help in material and process modifications to improve the performance as suggested in the recent articles [4-6]

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[4] M. Gollsch, S, Afflerbach, B.V. Angadi, M. Linder, Solar Energy 201 (2020) 810-818.
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