	Experiment title: In situ total scattering studies on alkali metal nitrate promoted MgO absorbents for CO_2 capture at moderate temperatures	**Experiment number**: MA-3415
Beamline: ID31	**Date of experiment**: from: 12.04.2017 to: 15.04.2017	**Date of report**: 19-09-2017
Shifts: 9	**Local contact(s)**: Blanco Maria	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Abdala Paula Macarena*[1], Dal Pozzo Alessandro*[1], Rekhtina Margarita*[1], Jakub Drnec*[2], Blanco Maria*[2] and Christoph Müller[1]

[1] Laboratory Energy Technology Laboratory of Energy Science and Engineering Leonhardstrasse 21, LEE P201 CH - 8092 ZUERICH

[2]Laboratory ESRF 71 avenue des Martyrs CS 40220 FR - 38043 GRENOBLE Cedex 9

Report:

The experiments led to the following publication:

Nanoscale, 2020, 12, 16462-16473

Effect of molten sodium nitrate on the decomposition pathways of hydrated magnesium hydroxycarbonate to magnesium oxide probed by in situ total scattering†

Margarita Rekhtina[a], Alessandro Dal Pozzo[ab], Dragos Stoian[c], Andac Armutlulu[a], Felix Donat[a], Maria V. Blanco[c], Zhu-Jun Wang[d], Marc-Georg Willinger[d], Alexey Fedorov[a], Paula M. Abdala[*a] and Christoph R. Müller[*a]

[a]Laboratory of Energy Science and Engineering, Department of Mechanical and Process Engineering, ETH Zürich, Leonhardstrasse 21, 8092 Zürich, Switzerland. E-mail: abdalap@ethz.ch; muelchri@ethz.ch

[b]Laboratory of Industrial Safety and Environmental Sustainability, Department of Civil, Chemical,

Environmental and Materials Engineering, Alma Mater Studiorum–Università di Bologna, Via Terracini 28, 40131 Bologna, Italy

[c]European Synchrotron Radiation Facility, 71 Avenue des Martyrs, Grenoble, France

[d]Scientific Center for Optical and Electron Microscopy, ETH Zürich, Auguste-Piccard-Hof 1, 8093 Zurich, Switzerland

Abstract

The effect of $NaNO_3$ and its physical state on the thermal decomposition pathways of hydrated magnesium hydroxycarbonate (hydromagnesite, HM) towards MgO was examined by in situ total scattering. Pair distribution function (PDF) analysis of these data allowed us to probe the structural evolution of pristine and $NaNO_3$-promoted HM. A multivariate curve resolution alternating least squares (MCR-ALS) analysis identified the intermediate phases and their evolution upon the decomposition of both precursors to MgO. The total scattering results are discussed in relation with thermogravimetric measurements coupled with off-gas analysis. MgO is obtained from pristine HM (N_2, 10 °C min^{-1}) through an amorphous magnesium carbonate intermediate (AMC), formed after the partial removal of water of crystallization from HM. The decomposition continues via a gradual release of water (due to dehydration and dehydroxylation) and, in the last step, via decarbonation, leading to crystalline MgO. The presence of molten $NaNO_3$ alters the decomposition pathways of HM, proceeding now through AMC and crystalline $MgCO_3$. These results demonstrate that molten $NaNO_3$ facilitates the release of water (from both water of crystallization and through dehydroxylation) and decarbonation, and promotes the crystallization of $MgCO_3$ and MgO in comparison to pristine HM. MgO formed from the pristine HM precursor shows a smaller average crystallite size than $NaNO_3$-promoted HM and preserves the initial nano-plate-like morphology of HM. $NaNO_3$-promoted HM was decomposed to MgO that is characterized by a larger average crystallite size and irregular morphology. Additionally, in situ SEM allowed visualization of the morphological evolution of HM promoted with $NaNO_3$ at a micrometre scale.

A second manuscript is currently in progress.