Project ID:

271511

Project acronym:

GaSTech

Project title:
Demonstration of Gas Switching Technology for Accelerated Scale-up of Pressurized Chemical Looping Applications (GaSTech)

Starting date of project:

1st of August 2017

Duration:

36 months

Deliverable D1.1,

Milestone 6

Involved Partners: ETH, SINTEF, ESAM

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WP N#	*Del/Mil N#*	Title	Contributors	Version	Lead beneficiary	Nature	Dissemina-tionlevel	Delivery date from contract	Actual delivery date
1	Mil 6	50 kg batch successfully delivered for GSR/GSPOx process	ESAM	1	ESAM	Material delivery	RE	02-2020	05-2020

Objective

This Deliverable/ milestone report is to act as an accompaniment to the materials for Gas Switching Partial Oxidation (GSPOx), delivered to SINTEF and NTU produced by ESAM.

The report summarizes the oxygen carrier (OC) basic production route and finished product analysis/characterization.

Please note the restricted nature of the content of this report, which should not be disclosed outside of the Consortium, as defined in the CA. If in doubt regarding the use of information contained in this report, please contact Euro Support Advanced Materials B.V.

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1 Description of deliverable/milestone

In deviation to the project plan, is was decided within the project consortium, to produce 50kg of sphere of an optimized GSR composition instead of 10kg of GSOP material, for which no working example has been found in literature. Also thermodynamic calculation done inside the project indicate that no such material exists today. The original delivery date for the 10kg of GSOP material was Feb. 2020. The delivery date of the 50kg of optimized spheres with the composition ($La_{0.80}Sr_{0.20}Fe_{0.95}Al_{0.05}O_{3+\delta}$) for GSR was May 2020.

The purpose of this deliverable is to summarize the material specifications, basic production routes and finished product analysis/characterization of the GSC oxygen carrier made by Euro Support. The manufacturing scale-up knowledge accrued from producing smaller batches is also deemed to be relevant for large scale/commercial production.

2 Material Specification

Material agreed to be produced: Lanthanum based oxygen carrier for GSR process.

	Finished Product	
Chemical Composition	**Target**	**Method of analysis**
La_2O_3	57 wt%	XRF (semi quantitative)
SrO	9 wt%	XRF (semi quantitative)
Fe_2O_3	33 wt%	XRF (semi quantitative)
Al_2O_3	1 wt%	XRF (semi quantitative)
Physical test methods		**Method of Analysis**
Tapped density	1.5-1.7 g/mL (solid spheres)	Quantachrome Autotap
Phase composition	>95% perovskite	XRD
Particle size	D10: 100 µm D50: 138 µm D90: 189 µm	Mastersizer 2000 (light scattering measurement)
Physical Characteristics		
Appearance	Dark grey sand like powder, free flowing	
Particle shape	Spherical (optical microscope)	
Crystal structure	Perovskite ($La_{0.80}Sr_{0.20}Fe_{0.95}Al_{0.05}O_{3+\delta}$)	

3 Production Route Overview

The 50kg amount oxygen carrier was prepared using industrially relevant equipment, located in Euro Support's production facility. This followed the production methodology outlined in the flowchart below (Figure 1) and used the equipment depicted and described in Figure 2 to **Error! Reference source not found.**4. For the preparation of the material produced for the GasTech project, the calcination conditions were optimized to achieve a calcined tapped density between 1.5 and 1.7/cm³. This is lower than previously obtain 1.901 gr/cm³ obtained for the material synthesized for milestone 3: $La_{0.85}Sr_{0.15}Fe_{0.95}Al_{0.05}O_{3+\delta}$. Whether the small difference in composition to the firstly produced $La_{0.85}Sr_{0.15}Fe_{0.95}Al_{0.05}O_{3+\delta}$ compared to the optimized $La_{0.80}Sr_{0.20}Fe_{0.95}Al_{0.05}O_{3+\delta}$ or a minor deviation in the chemical composition was responsible for the lower sintered density could not be determined.

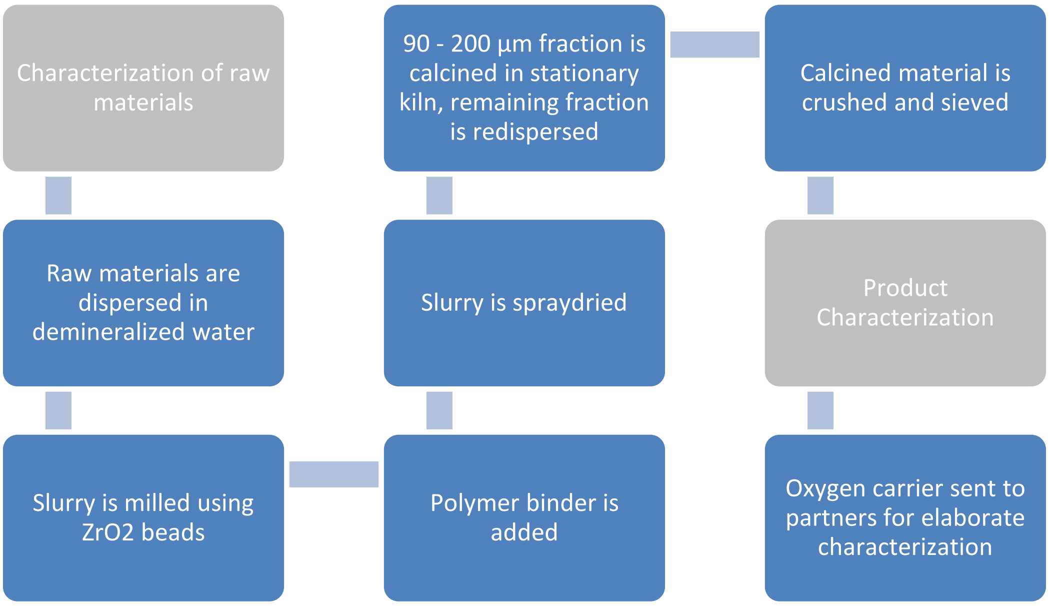

Figure 1: General methodology for producing the $La_{0.80}Sr_{0.20}Fe_{0.95}Al_{0.05}O_{3+\delta}$ oxygen carrier

Figure 2: Stationary Kiln (left) and ZrO2 bead mill (right)

Figure 3: Spray dryer

4 Sample analysis

4.1.1 $La_{0.80}Sr_{0.20}Fe_{0.95}Al_{0.05}O_3$ calcined spheres

4.1.2 Particle shape

Particle shape is evaluated by optical microscopy.

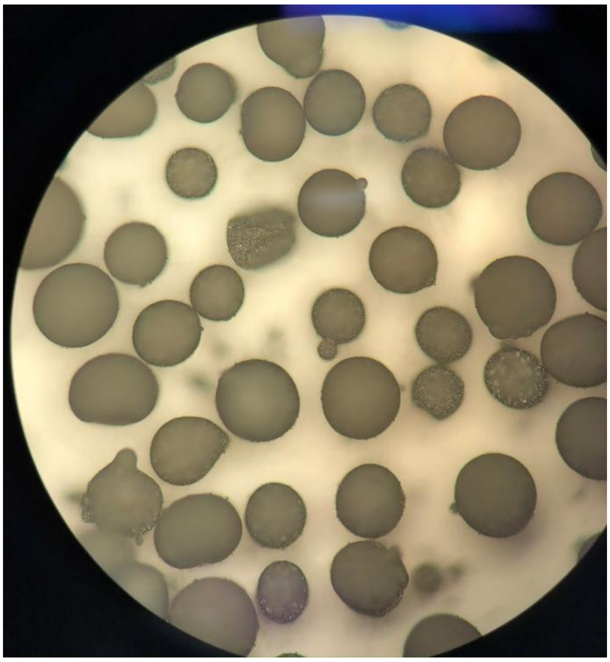

Figure 4: Calcined La0.80Sr0.20Fe0.95Al0.05O3 spheres

Most of the spheres appear to be solid and spherical; some bigger ones show indications that they are hollow.

A closer looks shows some porosity, which is required for the gas reactions. Calcining at higher temperature improved this marginally, however mechanical strength issues could not be resolved.

4.1.3 Particle Size

The sample is dispersed in water in between a monochromatic laser and a photodetector. The light scattering pattern that is measured by the photodetector is converted into a particle size distribution by a software algorithm.

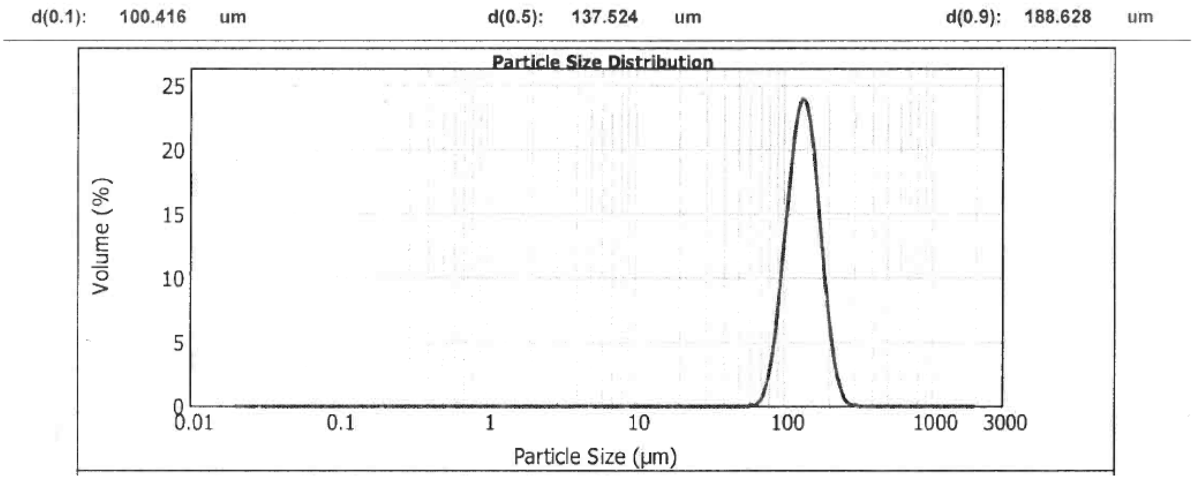

Figure 5: Particle size distribution of calcined La0.80Sr0.20Fe0.95Al0.05O3 spheres

4.1.4 Phase composition

XRD shows whether is material is sufficiently calcined, i.e. whether the raw materials are completely converted into the desired phase. A small impurity is present at 2theta=31 deg., however, this impurity disappears upon cycling (figure 8).

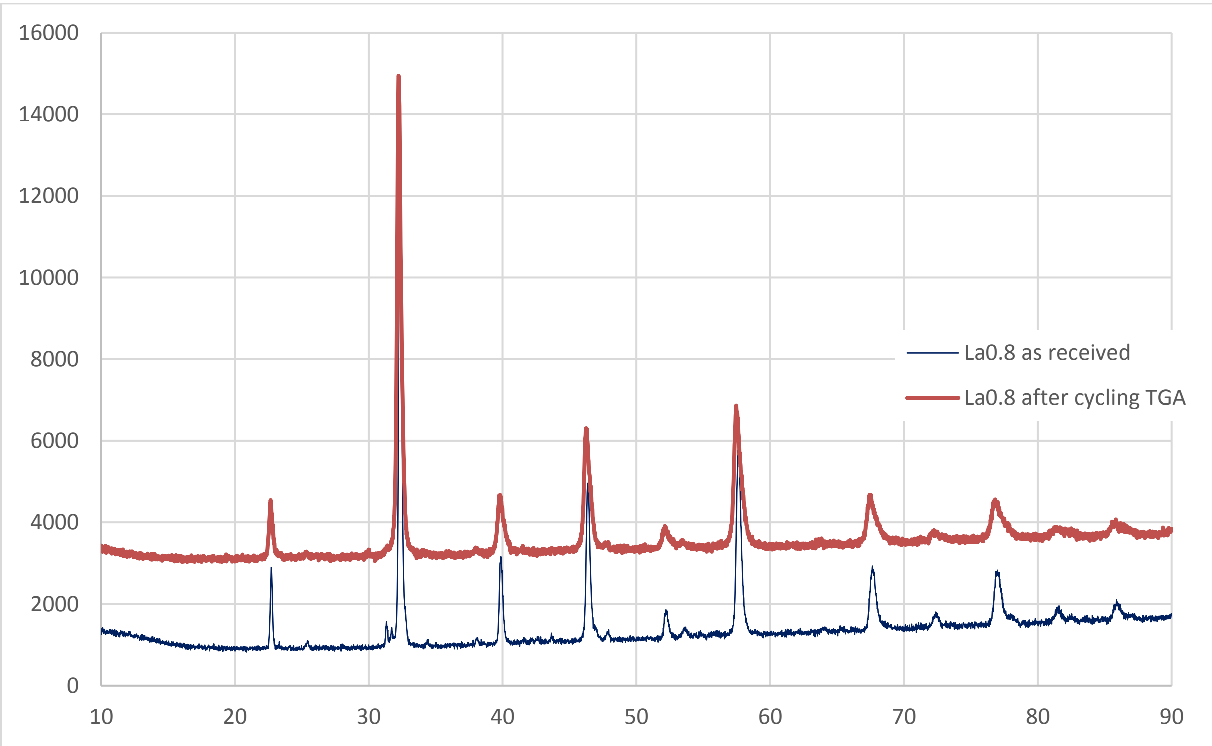

Figure 6: XRD spectrum of calcined La0.80Sr0.20Fe0.95Al0.05O3 spheres after sieving