

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 3

Involved Partners: ETH, SINTEF, ESAM

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| WP N# | Del/Mil N# | Title | Contributors | Version | Lead beneficiary | Nature | Disseminationlevel | Delivery date from contract | Actual delivery date |
|-------|------------|----------------------------------------------------------|--------------|---------|------------------|-------------------|--------------------|-----------------------------|----------------------|
| 1 | Mil 6 | 15 kg batch successfully delivered for GSR/GSPOx process | ESAM | 1 | ESAM | Material delivery | RE | 02-2020 | 08-2019 |

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

According to the original project plan, 15kg of material was delivered in August 2019.

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 ₂ O ₃ | 59 wt% | XRF (semi quantitative) |
| SrO | 7 wt% | XRF (semi quantitative) |
| Fe ₂ O ₃ | 33 wt% | XRF (semi quantitative) |
| Al ₂ O ₃ | 1 wt% | XRF (semi quantitative) |
| | | |
| Physical test methods | | Method of Analysis |
| Tapped density | 1.901 g/mL (solid spheres) | Quantachrome Autotap |
| Phase composition | >95% perovskite | XRD |
| Particle size | D10: 26 µm D50: 65 µm D90: 141 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 _{0.85} Sr _{0.15} Fe _{0.95} Al _{0.05} O _{3+δ}) | |

3 Production Route Overview

The 15kg 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.**

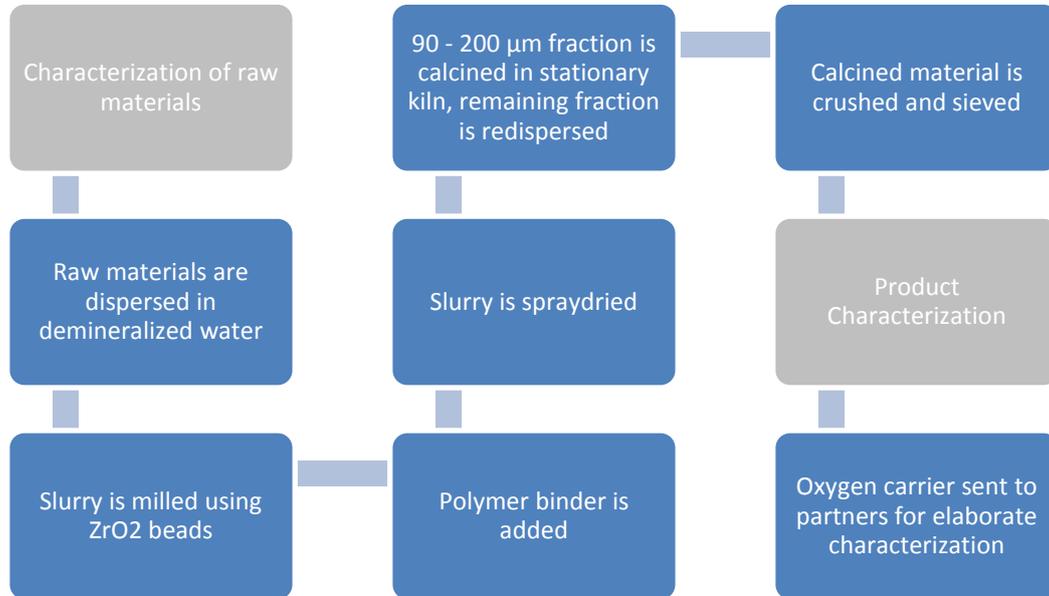


Figure 1: General methodology for producing the $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ oxygen carrier



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



Figure 3: Spray dryer

4 Sample analysis

4.1.1 $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ calcined spheres

4.1.2 Particle shape

Particle shape is evaluated by optical microscopy.

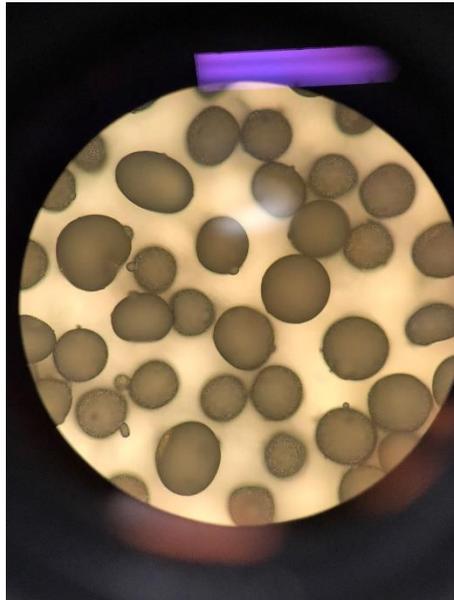


Figure 4: Calcined $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ spheres

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

A closer look shows some porosity, which is required for the gas reactions. This resulted in a generally low mechanical strength. 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.

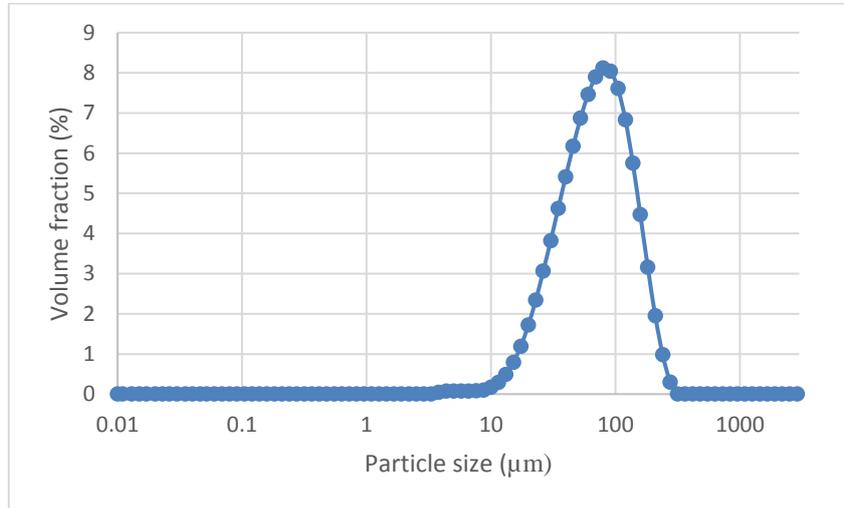


Figure 5: Particle size distribution of calcined $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ spheres 90-200µm fraction after sieving

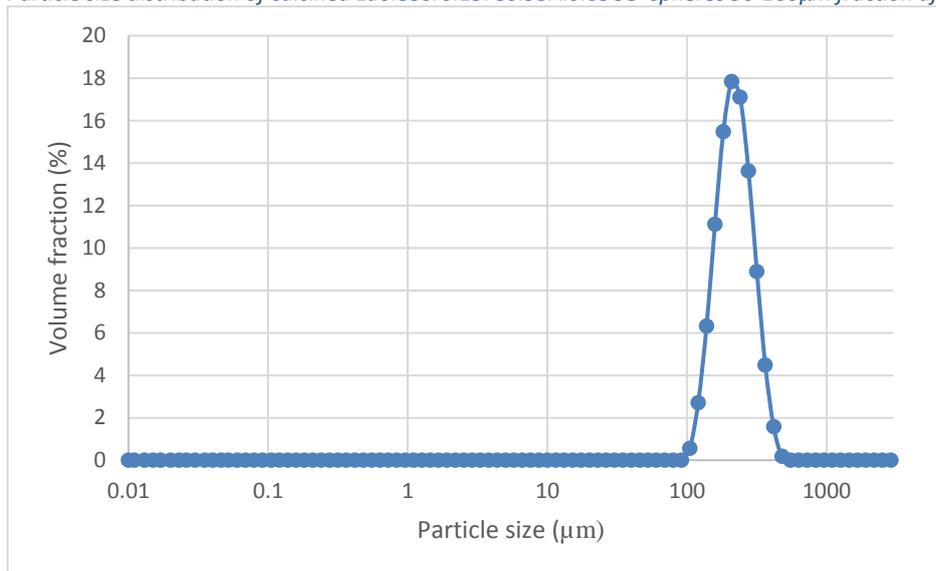


Figure 6: Particle size distribution of calcined $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ spheres, >200µm fraction after sieving

4.1.4 Phase composition

XRD shows the material is sufficiently calcined, i.e. the raw materials are completely converted into the desired phase (figure 7).

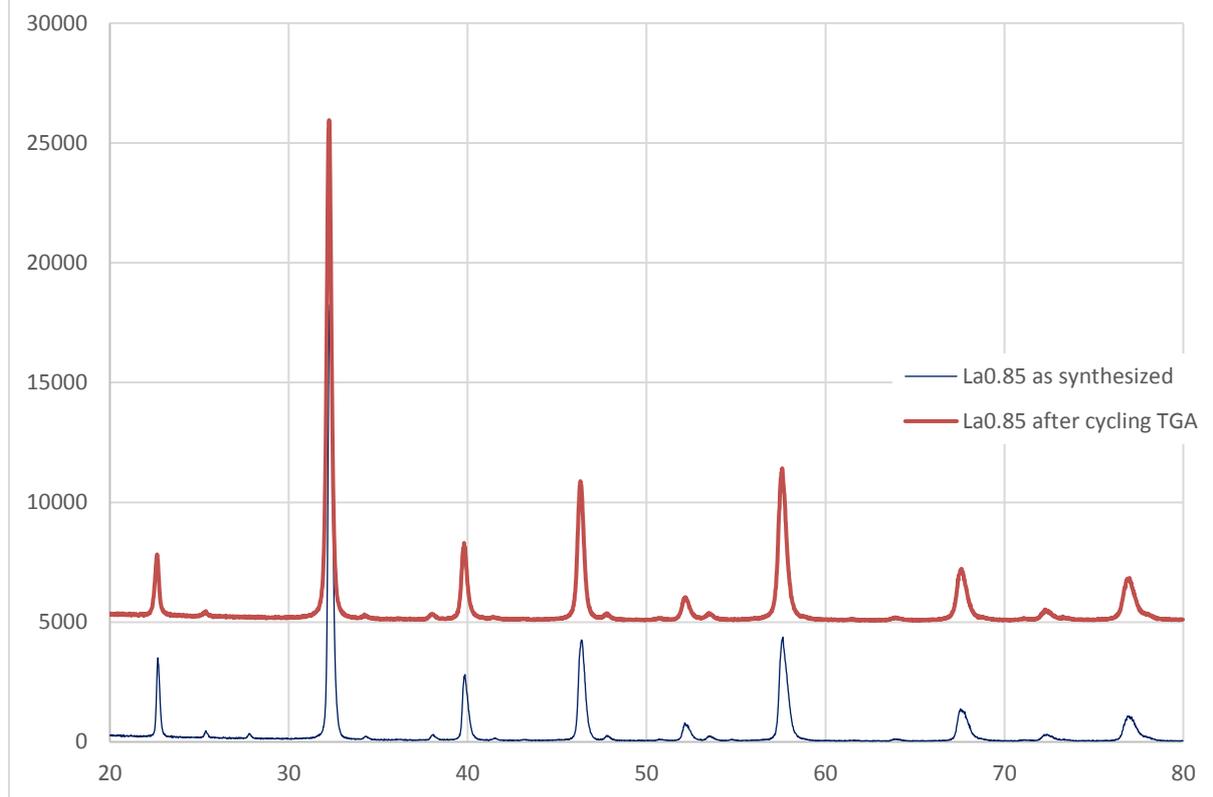


Figure 7: XRD spectrum of calcined $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$ spheres 90-200 μm fraction after sieving

(S)TEM analysis of the particles show the distribution of different elements within the particles. The analysis confirms homogeneous distribution of these elements within the particles.

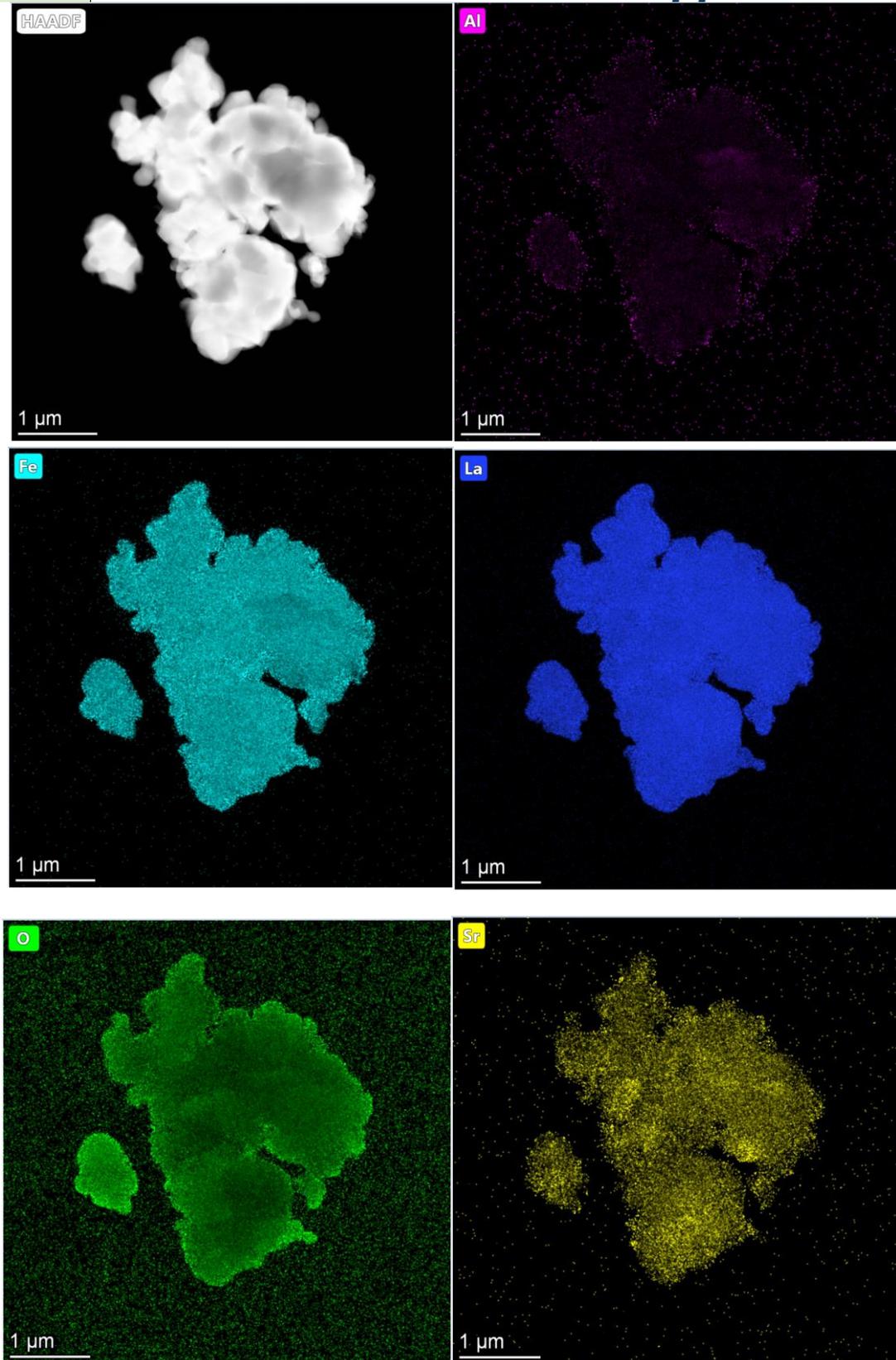


Figure 8: HAADF and EDX maps of $\text{La}_{0.85}\text{Sr}_{0.15}\text{Fe}_{0.95}\text{Al}_{0.05}\text{O}_3$