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 7

Involved Partners: ETH, SINTEF, ESAM

Author: Dr. Knuth Albertsen, Euro Support Advanced Materials B.V.

Contributions: Dr. Felix Donat, ETH Zürich

WP N#	*Del/Mil N#*	Title	Contributors	Version	Lead beneficiary	Nature	Dissemina-tionlevel	Delivery date from contract	Actual delivery date
1	Del 1.1	10* kg batch successfully delivered for GSC process	ESAM	1	ESAM	Material delivery	RE	31-03-2020	20-08-2018**
1	Mil 7	Production 10* kg sample of upgraded C28 oxygen carrier for the GSC tests	ESAM	1	ESAM	Report	RE	31-3--2020	7-12-201

*50 instead of 10kg produced and delivered on request of Sintef/NTU

** material delivered earlier in project due as Sintef /NTU need more time and material, for testing of GSC in cluster operation

Objective

This Deliverable/ milestone report is to act as an accompaniment to the materials for Gas Switching Combustion (GSC), delivered to SINFET 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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Description of deliverable/milestone

In deviation to the original project plan, 50kg of material were delivered in month 13 to Sintef and NTU instead of month 32. The reason for this early delivery is that the GSC reactor cluster will be ready soon and that material for trials is needed. The amount of materials needed has been increased from 10 to 50kg by Sintef/NTU.

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.

1 Material Specification

Material agreed to be produced: up-grades C28 oxygen carrier developed for the SUCCESS project..

	Finished Product	
Chemical Composition	**Target**	**Method of analysis**
Mn3O4	48 wt%	XRF (semi quantitative)
CaO	41 wt%	XRF (semi quantitative)
TiO2	8 wt%	XRF (semi quantitative)
MgO	3 wt%	XRF (semi quantitative)
Physical test methods		**Method of Analysis**
Tapped density	1.86 g/mL (solid spheres)	Quantachrome Autotap
Phase composition	>95% perovskite	XRD
Particle size	D10: 98 µm D50: 152 µm D90: 234 µm	Mastersizer 2000 (light scattering measurement)
Physical Characteristics		
Appearance	Dark grey sand like powder, free flowing	
Particle shape	Spherical (optical microscope)	
Crystal structure	Perovskite $Ca(Mn_{0.9}Ti_{0.1})O_{2.96}$ and MgO	

2 Production Route Overview

The 50kg target amount oxygen carriers were prepared using industrially relevant equipment, located in Euro Support's production facility. This followed the production methodology outlined in the flowchart below (**Error! Reference source not found.**) and used the equipment depicted and described in **Error! Reference source not found.** to Figure 4. The C28 oxygen carrier material was scaled up to the tonnage scale in the SUCCESS project. The tonnage scale produced material showed a relative high attrition rate, that could be attributed to a reduced calcination temperature used for the calcination of the bulk amount of the oxygen-carriers as an over calcination would have resulted in inactive material. For the preparation of the material produced for the GasTech project, the calcination conditions were optimized to achieve a calcined tapped density of 1.86g/cm³, which had given optimum results for smaller samples in the SUCCESS project.

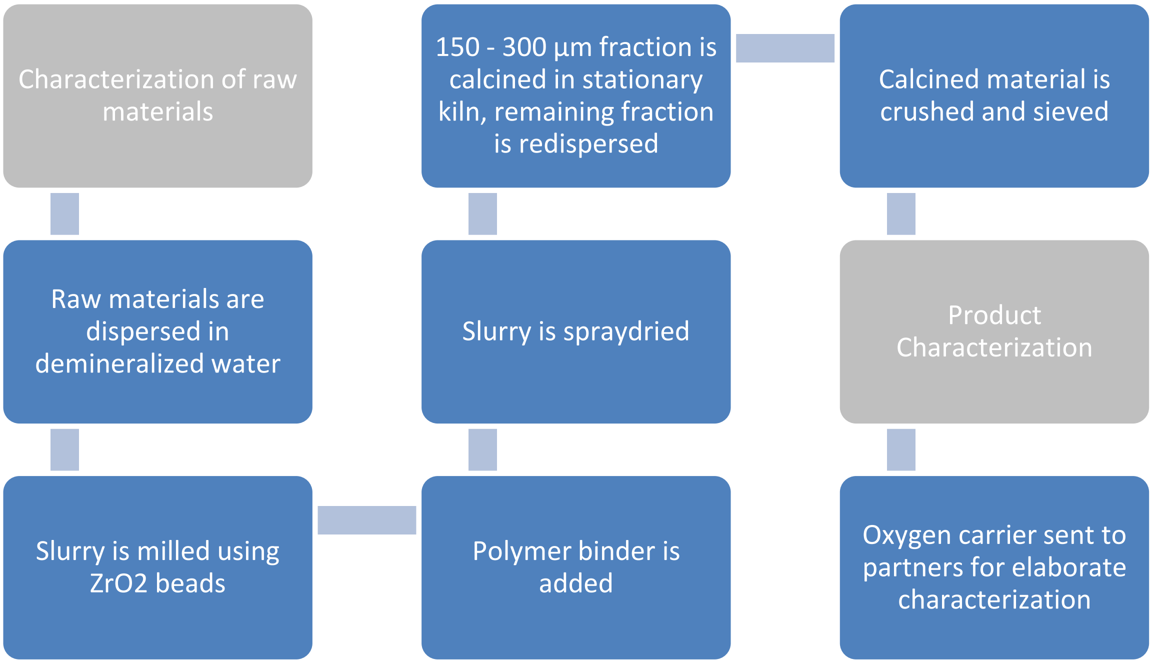

Figure 1: General methodology for producing the C28 oxygen carrier

Figure 2: Stationary kiln

Figure 3: ZrO2 bead mill

Figure 4: Spray dryer

3 Sample analysis

3.1 C28 calcined spheres

3.1.1 Particle shape

Particle shape is evaluated by optical microscopy and SEM microcopy.

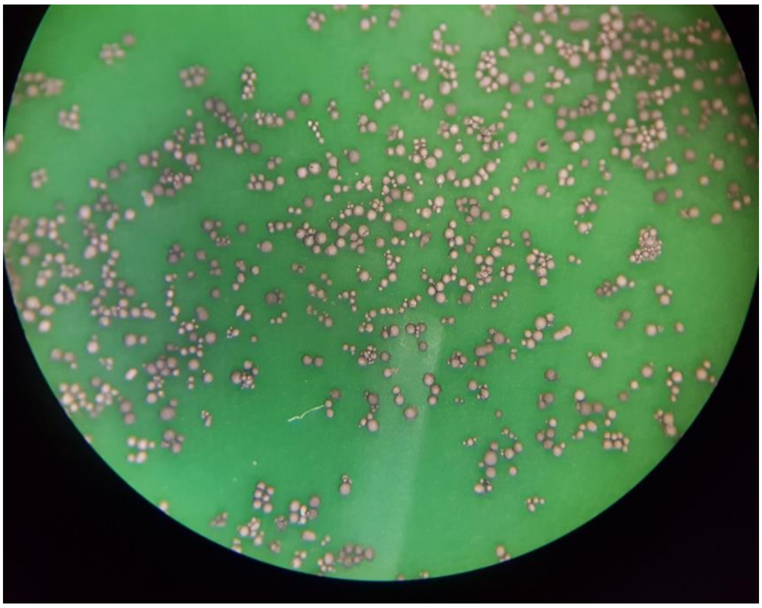

Figure 5 Calcined C28 spheres

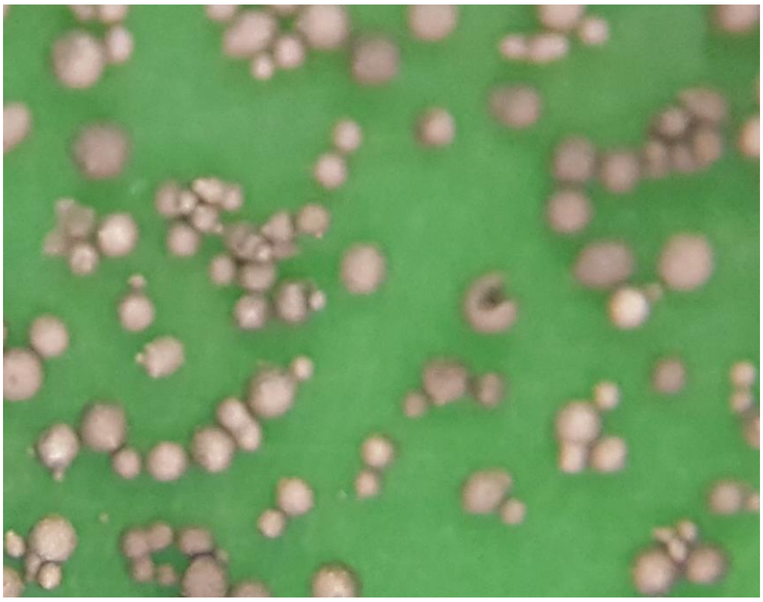

Figure 6 Calcined C28 spheres: Hollow bigger particle

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

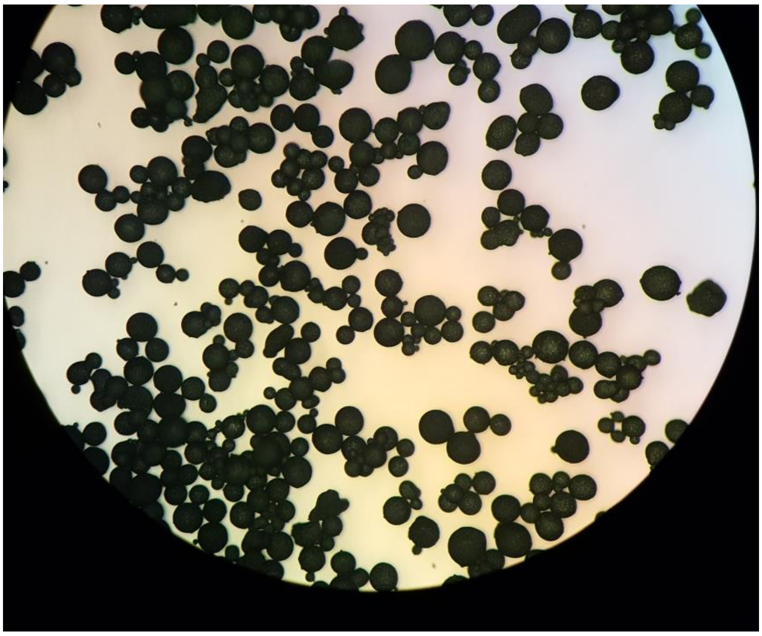

Figure 7 Calcined C28 spheres

The calcined spheres are homogeneously round and not aggregated.

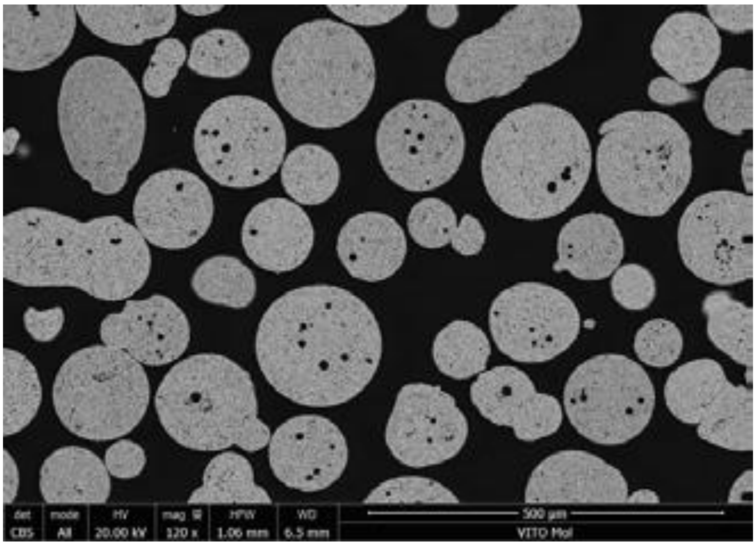

Figure 8 Solid interior of C28 spheres with little macro porosity (courtesy VITO)

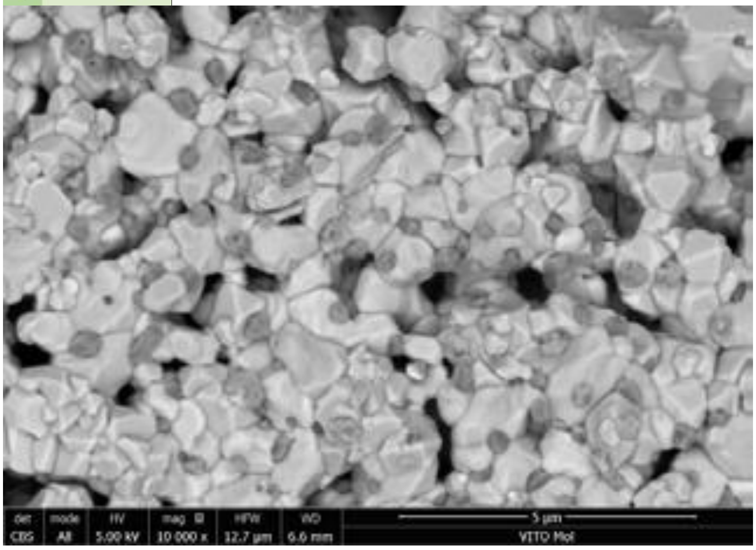

Figure 9 Porous microstructure of C28 spheres (courtesy VITO)

The spheres have a nearly fully filled internal structure (fig 8). A closer looks shows some porosity, which is required for the gas reactions. Spheres with a tapped density of up to 2.4g/cm³ show a very high mechanical strength, but only a very low oxygen exchange capacity as the material is nearly fully dense and the redox reactions are only controlled by diffusion and can only reach a part of fully sintered spheres, while the open porosity spheres show a improved reactivity as the reducing and oxidizing gases can directly interact also with the interior of the spheres.

3.1.2 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.

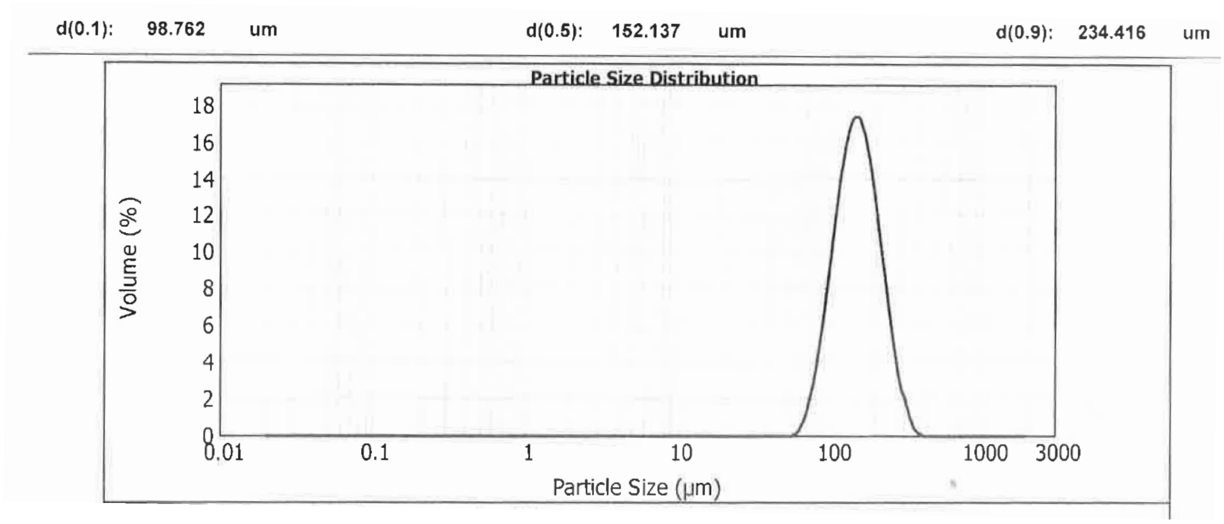

Figure 10 Particle size distribution of calcined C28 spheres

3.1.3 Phase composition

XRD shows whether is material is sufficiently calcined, i.e. whether the raw materials are completely converted into the desired phase. C28 oxygen carrier shows as main phase Perovkite type XRD pattern of the $Ca(Mn_{0.9}Ti_{0.1})O_{2.96}$ phase, as second phase free MgO could be detected.

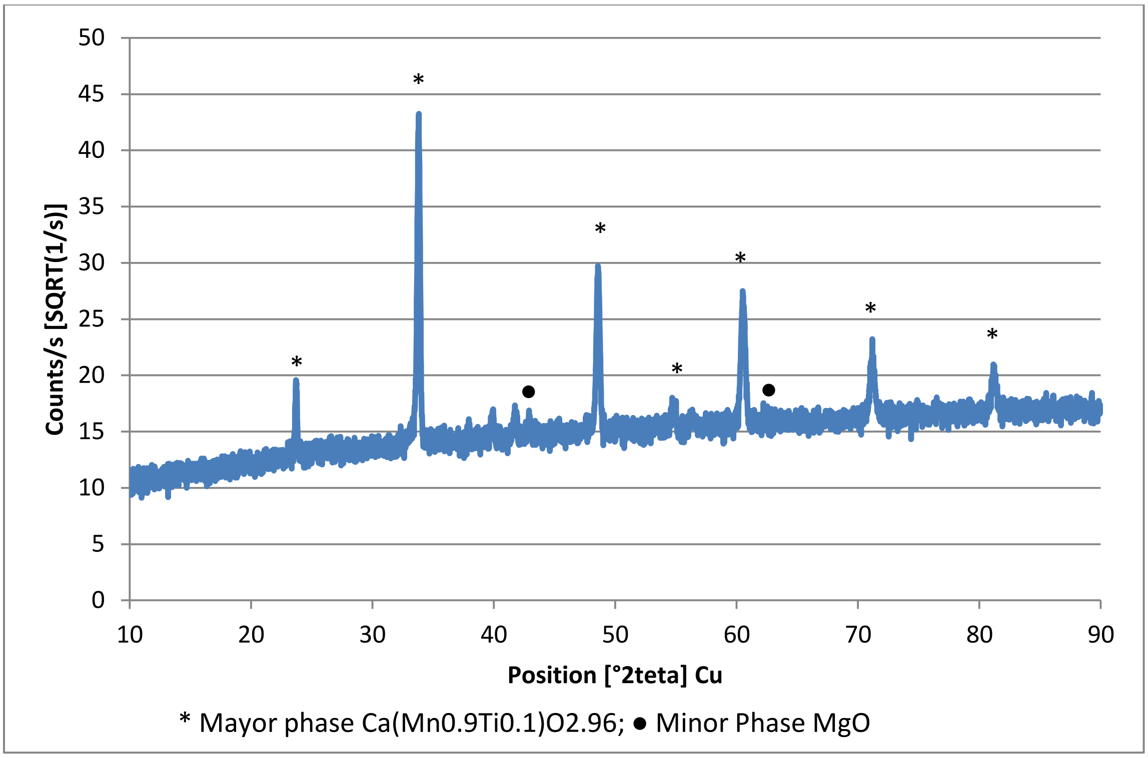

Figure 11 XRD pattern of calcined C28 spheres