**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 1

Involved Partners: ETH, SINTEF, ESAM

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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 first process | ESAM | 1 | ESAM | Material delivery | RE | 31-03-2018 | 04-04-2018*<br>05-05-2018** |
| 1 | Mil 1 | Production of 10 kg sample of oxygen carrier for the demonstration of GSWS | ESAM | 1 | ESAM | Report | RE | 30-4--2018 | 30-11-2018 |

* Delivery of first selected material

** Delivery of second material, optimized composition

**Objective**

This Deliverable/ milestone report is to act as an accompaniment to the 2 materials for Gas Switching Water Splitting (GSWS), delivered to SINFET and ETH, 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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Table of figures

Description of deliverable/milestone

The purpose of this deliverable is to summarize the material specifications, basic production routes and finished product analysis/characterization of the 2 materials for GSWS kg batch 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

In deviation to the original project plan, two materials instead of one material were produced on production equipment based on the initial development work performed at ETH and fine-tuned after the initial lab-scale process development at ESAM.

**First material agreed to be scaled up: Fe2O3 based oxygen carrier based on spinel raw material.**

| (70% Fe2O3 and spinel based spheres) Finished Product | | |
|---|---|---|
| | | |
| **Chemical Composition** | **Target** | **Method of analysis** |
| Fe2O3 | 70 wt% | XRF (semi quantitative) |
| Al2O3 | 17.4 wt% | XRF (semi quantitative) |
| MgO | 4.6 wt% | XRF (semi quantitative) |
| CuO | 8 wt% | XRF (semi quantitative) |
| | | |
| **Physical test methods** | | **Method of Analysis** |
| Particle shape | solid and hollow spheres | Optical microscopy |
| Phase composition | 2 phases, spinel and Fe-spinel | XRD |
| Particle size | D10: 74 µm<br>D50: 151 µm<br>D90: 297 µm | Mastersizer 2000 (light scattering measurement) |
| | | |
| **Physical Characteristics** | | |
| Appearance | Dark grey sand like powder, free flowing | |

**Optimized composition to be scaled: Fe2O3 based oxygen carrier based with optimized spinel structure**

| (Optimized composition single phase spinel ) Finished Product | | |
|---|---|---|
| | | |
| **Chemical Composition** | **Target** | **Method of analysis** |
| Fe2O3 | 70.3 wt% | XRF (semi quantitative) |
| Al2O3 | 5.0 wt% | XRF (semi quantitative) |
| MgO | 19.7 wt% | XRF (semi quantitative) |
| CuO | 5.0 wt% | XRF (semi quantitative) |
| | | |
| **Physical test methods** | | **Method of Analysis** |
| Particle shape | solid and hollow spheres | Optical microscopy |
| Phase composition | single phase Fe spinel | XRD |
| Particle size | D10: 73 µm<br>D50: 149 µm<br>D90: 296 µm | Mastersizer 2000 (light scattering measurement) |
| | | |
| **Physical Characteristics** | | |
| Appearance | Dark grey sand like powder, free flowing | |
| Particle shape | Hollow spheres Spherical (optical microscope) | |

# 2 Production Route Overview

The two times 25 kg target quantity of GSWS oxygen carrier were prepared using industrially relevant equipment, located in Euro Support's production facility. This follows the production methodology outlined in the flowchart below (Figure 1) and uses the equipment depicted and described in **Error! Reference source not found.** to Figure 4.

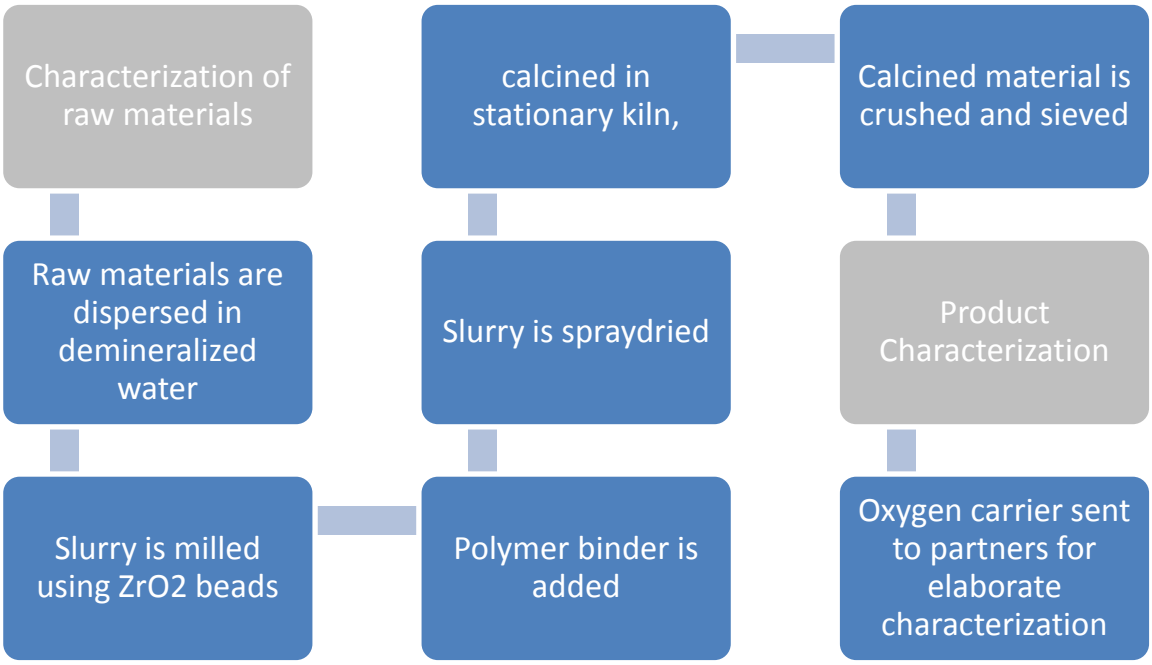


*Figure 1: General methodology for producing the GSWS oxygen carrier*


Figure 2: Stationary kiln


Figure 3: ZrO2 bead mill


Figure 4: Spray dryer

# 3 Sample analysis

## 3.1 70% Fe2O3 and spinel based spheres

### 3.1.1 Particle shape

Particle shape is evaluated by optical microscopy and SEM microcopy

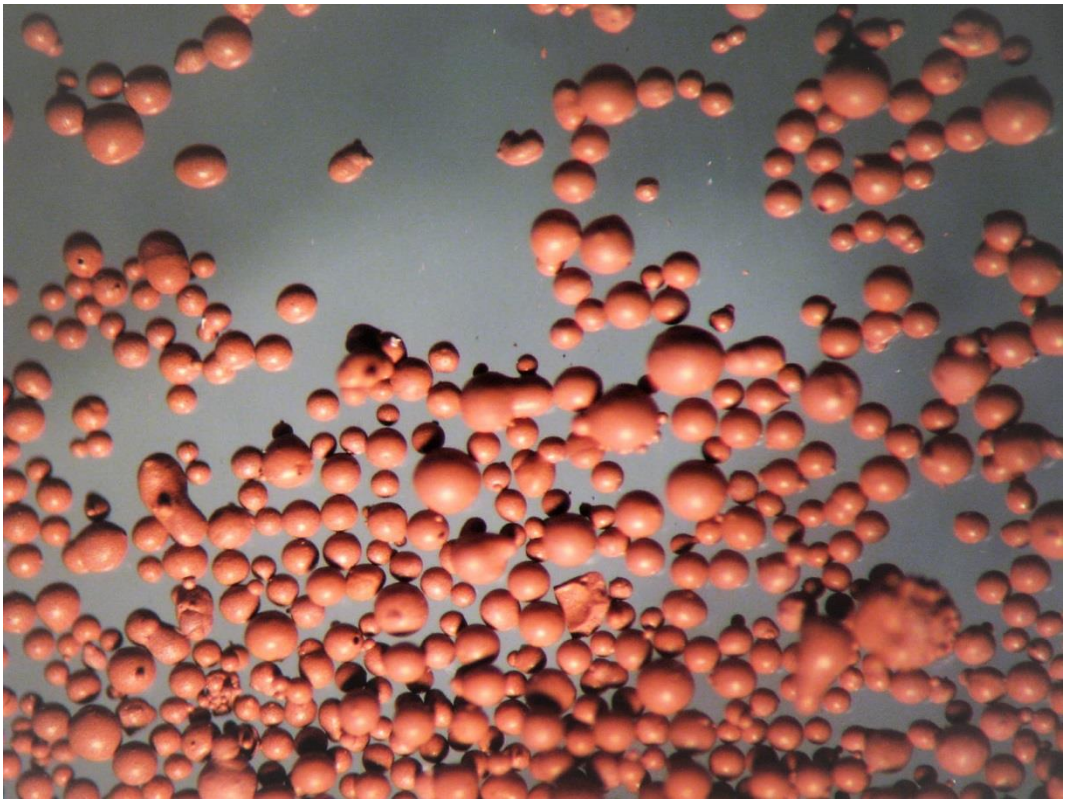

*Figure 5 uncalcined spheres after spray drying 70% spinel based composition*

*Figure 6 Calcined spheres70% spinel based composition*

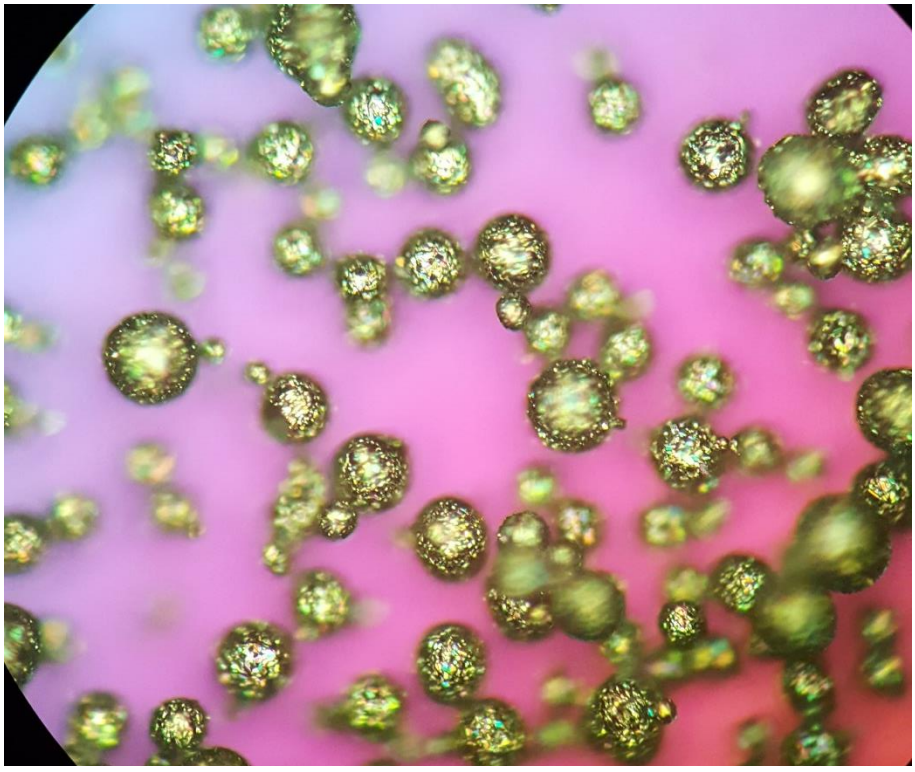

*Figure 7 Calcined spheres 70% spinel based composition*

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

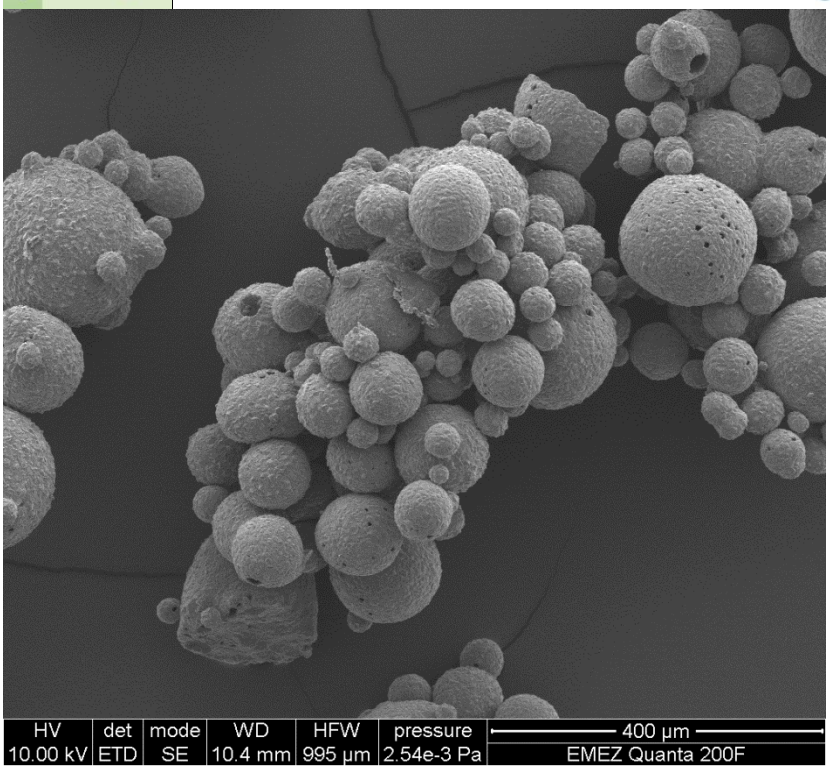


*Figure 8 Calcined spheres 70% spinel based composition SEM picture, bigger spheres seem to be hollow*

The average particle size of the produced spheres is 151µm.

### 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.

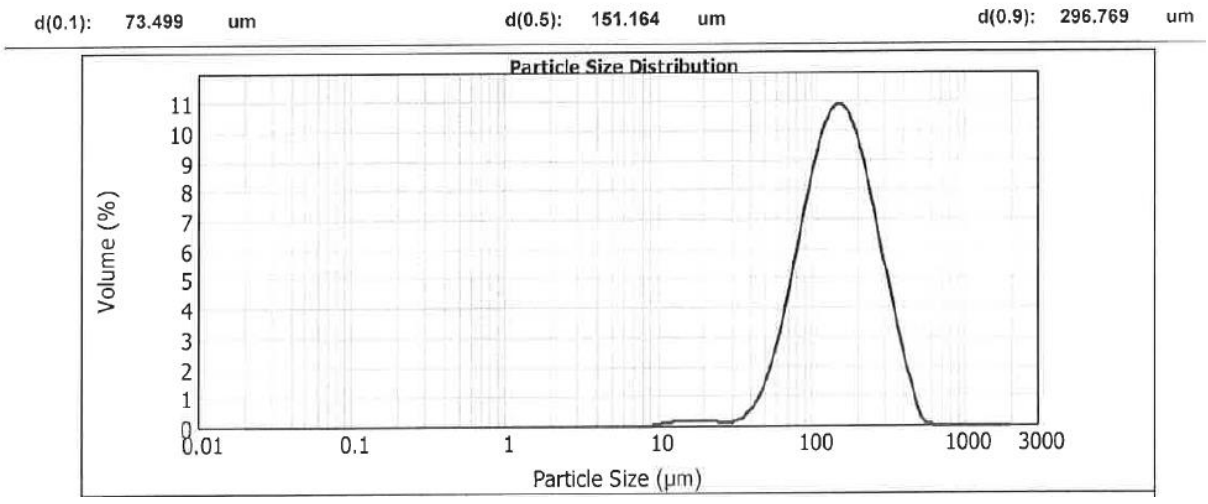


*Figure 9 Particle size distribution of calcined spheres70% spinel based composition*

### 3.1.3 Phase composition

X-Ray Diffraction is an analytical technique in which the diffraction pattern of an X-ray beam is measured. This diffraction pattern is characteristic for the crystallographic composition of the sample.

XRD shows whether is material is sufficiently calcined, i.e. whether the raw materials are completely converted into the desired phase. The 70% $Fe_2O_3$ spinel based materials show after calcination the pattern of pure spinel $MgAl_2O_4$, the spinel $Mg(Fe_{0.5}Al_{0.5})_2O_4$ and $Fe_2O_3$ are detected. After redox cycling experiments at high temperature, the spinel phase $Mg(Fe_{0.5}Al_{0.5})_2O_4$ is the dominant phase.

## 3.2 Optimized composition with 70% $Fe_2O_3$ content

### 3.2.1 Particle shape

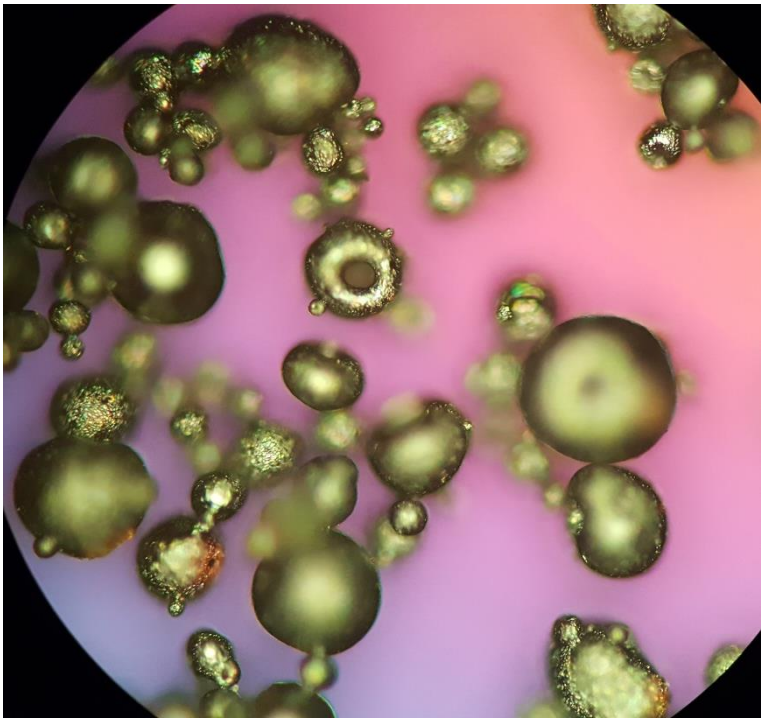

*Figure 10 calcined spheres of optimized composition*

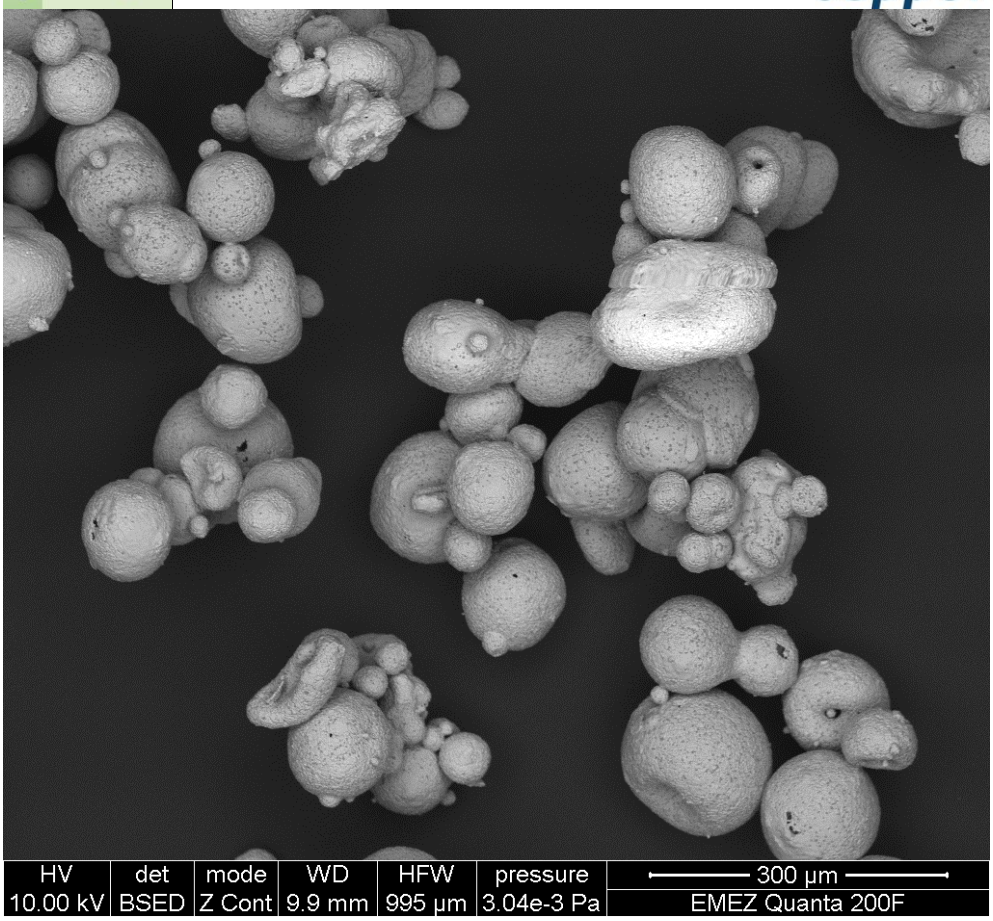


*Figure 11 Particle shape optimized composition SEM picture*

The shape of the particles is more donut like compared to the 70% spinel based composition.

### 3.2.2 Particle Size

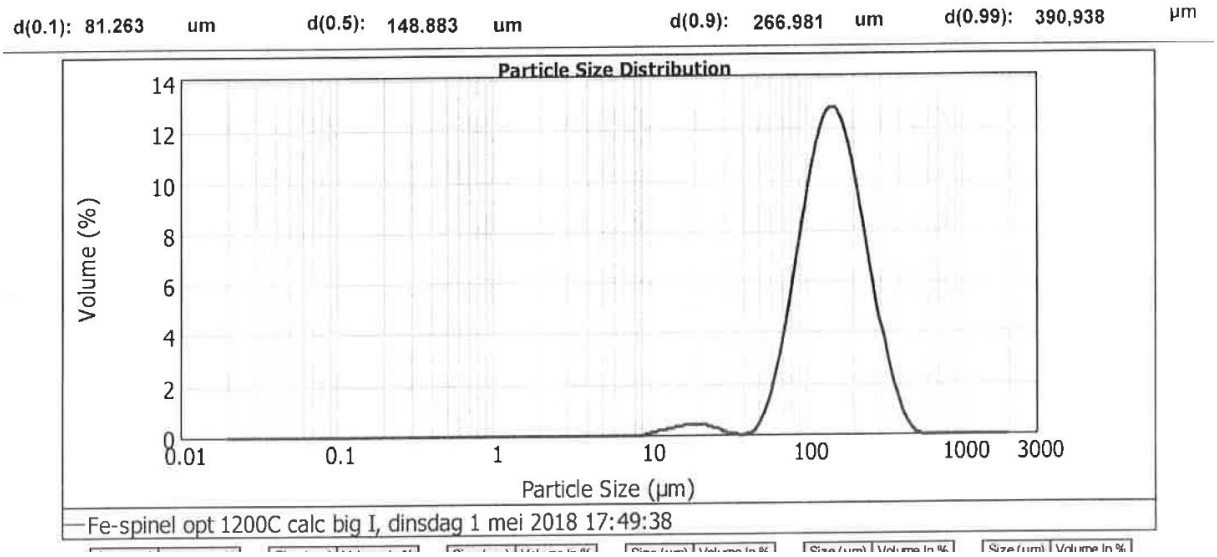


*Figure 12 The particle size distribution is very similar to the other GSWS oxygen carrier.*

The average particles size of the produced spheres is 149µm.

### 3.2.3 Phase composition

XRD shows whether is material is sufficiently calcined, i.e. whether the raw materials are completely converted into the desired phase. The optimized composition shows a single phase spinel type XRD pattern, $Mg(Fe_{0.9}Al_{0.1})_2O_4$. XRD analysis after redox cycling confirmed that this spinel phase was the only crystalline phase in the material, suggesting a high chemical stability of this particular oxygen carrier.