CCSP WP5.2.1, D546
2015

Arshe Said
Mika Järvinen

Reporting of results obtained with Slag2PCC pilot-scale test facility unit at Aalto

ccsp
Carbon Capture and Storage Program

CLEEN LTD
ETELÄRANTA 10
P.O. BOX 10
FI-00130 HELSINKI
FINLAND
www.cleen.fi

ISBN XXX-XX-XXXX-X
ISSN XXXX-XXXX

ccsp
Carbon Capture and Storage Program

CLEEN
Cluster for Energy and Environment

Aalto University
CCSP WP5.2.1, D546

Arshe Said & Mika Järvinen

Reporting of results obtained with Slag2PCC pilot-scale test facility unit at Aalto

Aalto University
February 2015

A Said, M Järvinen

Report Title: D546 – Reporting of results obtained with Slag2PCC pilot-scale test facility unit at Aalto

Keywords: precipitated calcium carbonate, Slag2PCC, particle morphology

Abstract

The work at Aalto University has recently focused on steering the particle quality of the precipitated calcium carbonate product. Additional lab-scale test were preformed to investigate the effect of carbonation temperature on the morphology of PCC. We also conducted some pilot scale test in which we investigated the solvent recyclability.

Results from both lab-scale and pilot scale tests

1. Lab-scale tests

We important to investigate the effect of process parameters on the PCC quality in a lab-scale priori the pilot scale. For this reason, it was decided to investigate the effect of temperature at lab-scale.

1.1 Effect of temperature on PCC morphology

The effect of carbonation starting temperature on the morphology of PCC precipitated via the Slag2PCC process was investigated in a 5L jacketed glass reactor by performing carbonation tests over a wide range of temperatures from 5°C to 80°C. The calcium-rich solution was prepared using 1M of NH_4Cl and 100 g/l slag to liquid ratio during the calcium extraction process. The solution volume (4L), agitation rate (800rpm) and CO_2 flow (0.4 L/min) was the same for each test. The initial carbonation temperature was set using a water bath in which the water could be cooled with ice or electrically heated.

The PCC product was collected washed and vacuum filtered. SEM images selected tests are shown in Fig. 1. At temperatures between 5 °C to 25°C the primary particle morphology is rhombohedral calcite. At temperatures between 45°C to 50°C the particles lose their rhombohedral character and become less regular and more clumpy while still remaining calcite. First at 60°C aragonite was seen to form, with the product becoming nearly pure aragonite at 80°C.

In addition to the SEM micrographs, samples from selected tests were also analysed for particle size distribution (PSD). The results of this analysis are shown in Fig 2. The mean size (d_{50}) of the particles after introduction of ultra sound (US) appeared to increase with temperature from 27µm at 5°C up to a maximum of 57µm at 45°C. Increasing the temperature leads to smaller particle sizes. The aragonite particles produced in the 60°C and 80°C tests were in the size range of 1-10µm.

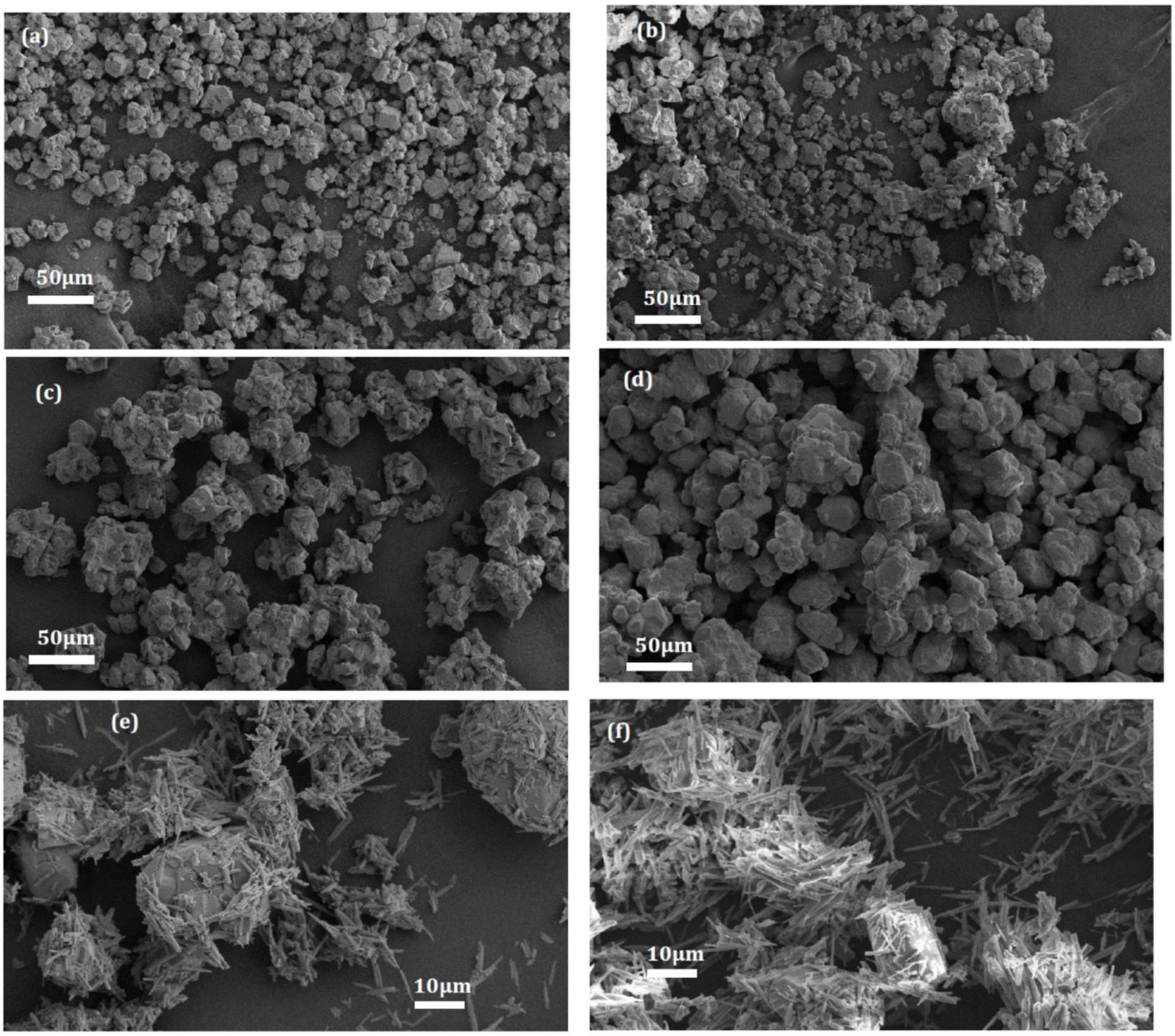

Figure 1. Scanning electron microscope (SEM) pictures of the produced PCC particles. a) 5 °C b) 25°C, c) 45°C, d) 50°C, e) 60°C, and f) 80°C

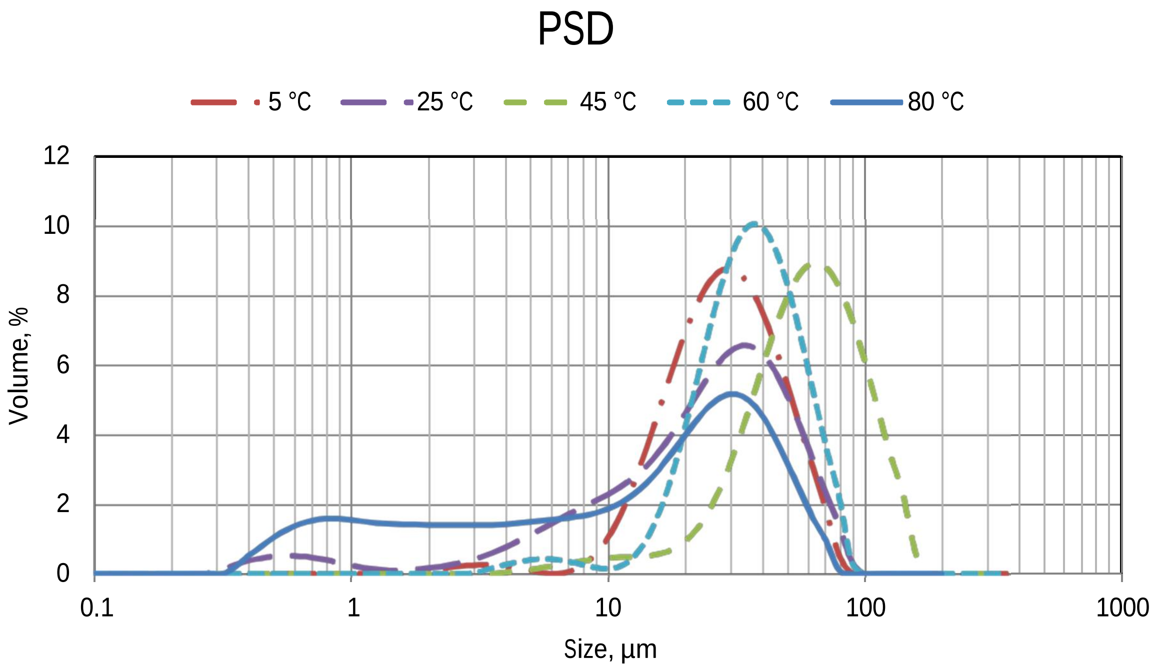

Figure 2. PSD of PCC at temperatures

2. Pilot-scale tests

2.1. Solvent recyclability tests

The preliminarily solvent recyclability (Fig. 3) results showed that fresh and recycled solvent has almost the same Ca extraction efficiency. The highest Ca-extraction efficiency achieved was 78-% by the fresh solvent and 60% as the lowest extraction efficiency during the second recycle test (Cycle-2). There is some fluctuation in the results that is due to the difficulties taking samples from a 200 L reactor.

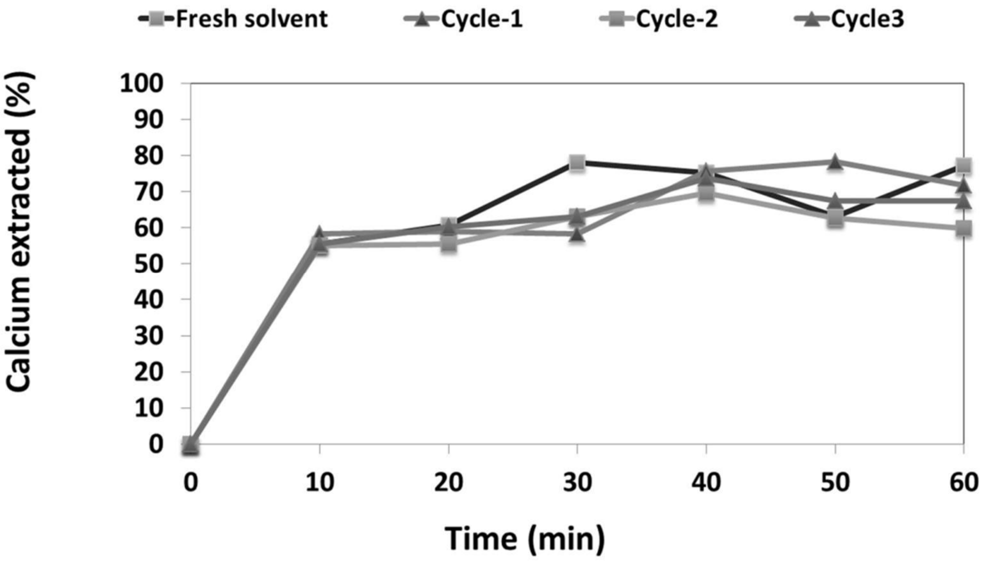

Figure 3. Solvent recyclability tests

Conclusions

- At temperatures of 25°C and below, the primary particle morphology is rhombohedral calcite. From 45°C to 60°C the particles become less rhombohedral in shape, less regular, and clumpier while still remaining calcite.
- At temperatures of 60°C aragonite (<10μm) began to precipitate with the product becoming nearly pure aragonite at 80°C.
- At 50°C some aragonite or calcite morphology was produced
- All particles showed some degree of agglomeration.
- Primary particle size seemed to have a minimum at 5°C and increase with increasing temperature, though particle size distribution (PSD) analysis by laser sizing with application of ultrasound (6 minutes) to break agglomerates would be a better way to confirm this.
- The highest Ca-extraction efficiency achieved from the pilot plant was 78%. The grain size of the feed slag was < 250μm
- Preliminarily solvent can be recycled at least three times