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Tests with sub-demo for Slag2PCC at Åbo Akademi University

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Report Title: D511 – Tests with sub-demo for Slag2PCC at Åbo Akademi University

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Abstract

In order to progress further with the mineral carbonation process concept “slag2PCC” developed at Aalto University, a small-scale (~75 liters) reactor setup has been constructed at Åbo Akademi University during CLEEN CCSP FP1-2. Previous research has mainly concentrated on determining the most beneficial stepwise reaction conditions for efficient calcium extraction from slag to aqueous solutions and further precipitation as solid precipitated calcium carbonate (PCC). Based on these studies, details like the best solvent, concentration of the solvent, solid-to-liquid ratio, residence times in the reactors etc. have been assessed.

In the current work the focus has been on detailed process design instead of studying the process steps separately. The goal has been a setup that can be operated both batchwise and continuously, and that can be used to determine whether the small-scale results are applicable also for a larger system.

One of the aspects that have not been addressed previously are the removal of solid matter from the aqueous streams, considering both slag residue after extraction and PCC product after carbonation. Also, product quality as such, including particle size, crystal morphology and product purity, is a topic requiring more attention, since in small scale batch experiments large variations have been unavoidable. Moreover, equipment related to process measurement and control such as pH and temperature meters have been studied. All measures have also targeted at efficient regeneration of the process solvent to minimize the need for make-up chemicals.

Summary of the current work

The research work discussed in this report has been extensively summarized in various publications available via the CLEEN portal (see references). Thus, only the main findings and the current status of the work are presented here, as well as guidelines for future work.

The process concept comprises a two-step system, where calcium is first selectively extracted from steel converter slag in one reactor after which the aqueous ions are contacted with dissolved carbon dioxide in another reactor to produce precipitated calcium carbonate (PCC). Several solvents were studied earlier [1], and ammonium chloride was found to have good properties, ensuring both efficient and selective extraction, but also a sufficient precipitation yield. [2] It was found to be beneficial to limit the solvent strength to 1 mol/L at maximum, since at higher concentrations also other elements were extracted from the slag. Also, slag-to-liquid ratios below 100 g/L were recommended, since otherwise the extraction efficiencies would be noticeably reduced. [3-5] Residence times have also been analyzed and reported earlier. [2]

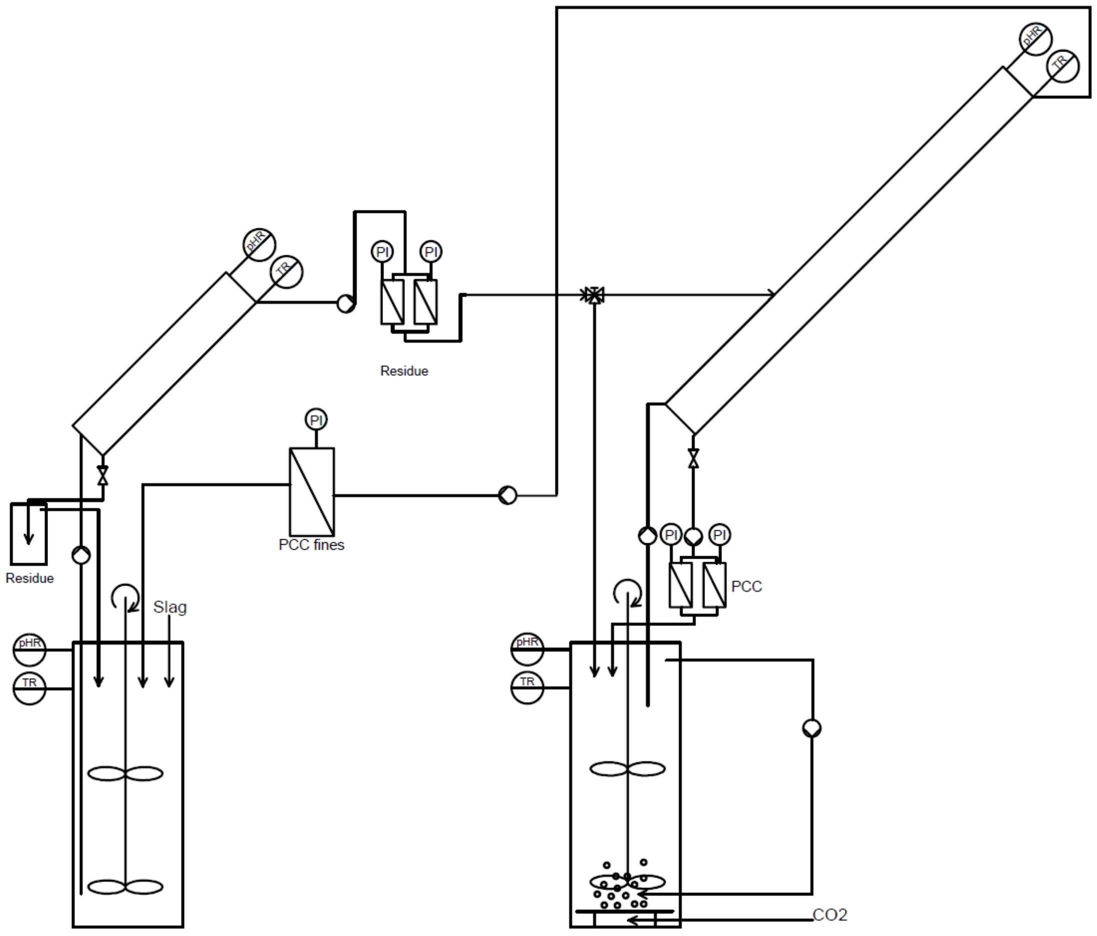

Figure 1. A schematic presentation of the constructed demo setup for production of PCC from steel converter slag.

Figure 1 represents the constructed reactor setup in detail. On the left the extraction reactor is presented and on the right the carbonation reactors. For separation of solid matter a combination of so-called inclined settlers and conventional 1µm filters is used, separately for the slag residue and for the precipitated carbonates. The principle and preliminary experiments with inclined settlers are discussed by Filppula [6]. The carbonation step is divided into two reactors, since that was found to be a reasonable method to adjust the composition and pH of the recirculating solvent stream. [5, 7] In Figure 2 the laboratory setup is shown with reactor lids visible at the front. Reactor vessels are presented in Figure 3. The design principles of this setup are discussed more thoroughly in [4].

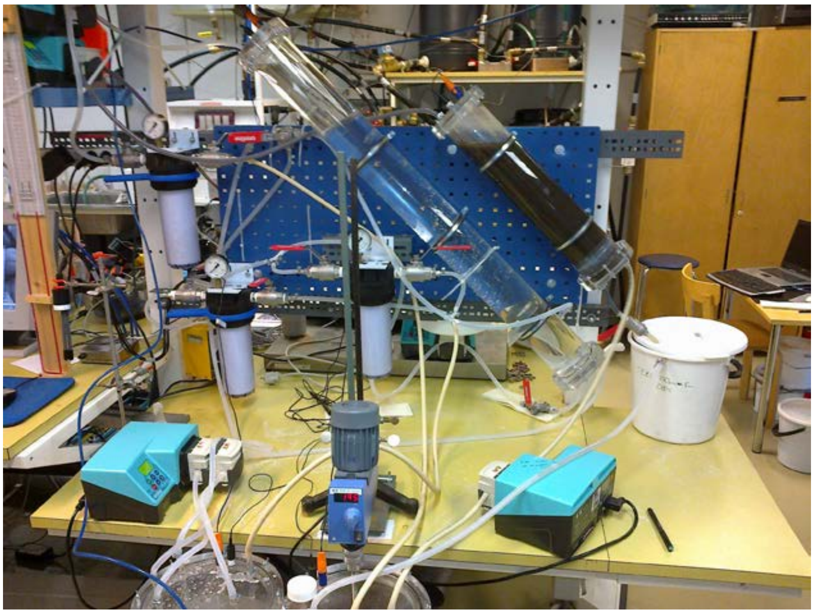

Figure 2. The laboratory sub-demo setup. (January 2013)

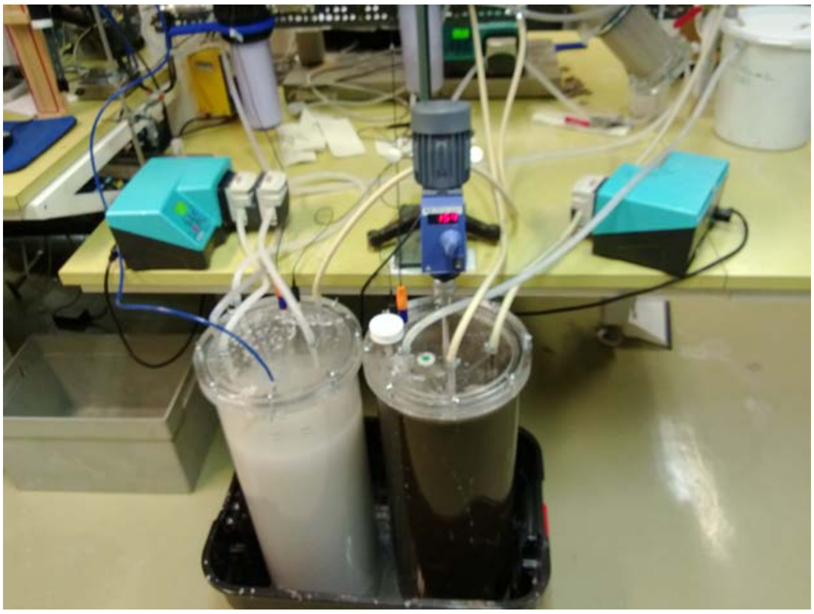

Figure 3. Reactors of the sub-demo setup. (January 2013)

The main target of the research is to verify whether a good quality PCC can be manufactured by a continuous process by using steel slag as a calcium source. The driving force for developing a continuous setup is the varying composition of steel converter slag. By following the pH and temperature in the various reactors as shown in Figure 1, and by adjusting the solid and liquid flow rates accordingly it should be possible to maintain approximately constant conditions in the reactors.

So far, only one experiment has been completed with the setup. Besides the need of some practical changes to ensure a smooth operability, no surprises were encountered. The amount of PCC product remained low, only 215 g from 4200 g steel converter slag, but this can be explained by the fact that the experiment had to be cancelled after one hour due to leakage problems before the carbonation had proceeded further. Based on the batch tests the expected yield of carbonate would have been between 1-1.5 kg. Even though the precipitate had a greyish color due to the mentioned leakage, in SEM analysis only minor impurities were detected, mainly silicon, chlorine and aluminum. The morphology of the crystals varied between the spherical vaterite and cubical calcite. These first results have been reported in [4, 8]

Figure 4. Produced PCC (left) and residual slag (right) from a continuous experiment run with the demo setup.

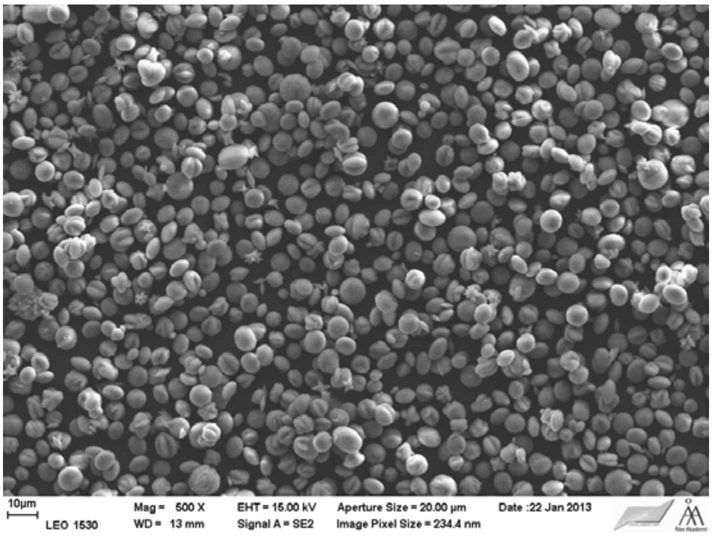

Figure 5. Produced carbonate from the PCC settler unit after a 60 min carbonation time.

Currently, modification of the reactor setup is ongoing, the target being to obtain results from a longer run shortly. Then, also the product properties as well as pH and temperature conditions in the reactors can be analyzed further.

Note that several related aspects and details on water processing, liquid/solid separations and analysis methods are reported in deliverable D512 [9].

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