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Report on results obtained with slag2pcc demo

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Report Title: Report on results obtained with slag2pcc demo

Key words: pilot-scale test facility, equipment, testing

Abstract

The Slag2PCC process route aims at converting calcium containing industrial by-products into valuable precipitated calcium carbonate (PCC) product. During CCSP1 the slag2pcc concept was taken from a two stage batch process to a continuously operating process that produces good quality PCC while the design parameters for larger scale were determined. The object of the phase 2 was to continue the work on the process design and cost estimate of the pilot-scale test facility. In addition, construction was set to begin during phase 2.

The pilot-scale design was completed in 2012, followed by ordering of the equipment. One of the challenges was that all the equipment in the markets are either designed for the laboratory level or for the industrial level, thus it was difficult to find suitable equipment for the test facility. However, the construction work began in January 2013.

The first set-up of the pilot-scale test facility was ready in January in 2014. Next, a series of simplified tests using only water were conducted. After the found leaks were repaired, the test was done with water and steel slag. This revealed that the set filters were not enough, thus a qualitatively filter of 0.45 µm was added resulting with very clean filtrate solution. These tests and repairs were followed with tests using aqueous solution of ammonium salt and steel converter slag. The produced precipitate was slightly yellow. The gas distribution frame was likely the cause of problem (welding was rusty), thus new one has been ordered and should arrive at any day. The data logger problems that occurred during the tests have been repaired. The real experimental work is going to start soon.

Espoo, April 2014

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1 Introduction

Fixation of CO_2 to mineral matter offers an alternative for geological CO_2 storage. This topic has been studied in Finland for more than a decade on laboratory scale and results have encouraged continuing development work towards piloting and demonstration. The Slag2PCC process route aims at converting calcium containing industrial by-products (with main focus on steel converter slag) into valuable precipitated calcium carbonate (PCC) product. In this method, an aqueous solution of ammonium salt is used to extract calcium from the by-product/waste material. After removal of the solid residue, CO_2 is bubbled through the solution producing stable calcium carbonate as an end product. Ammonium salt solution is recovered simultaneously and can thus be reused.

During CCSP1 the slag2pcc concept was taken from a two stage batch process to a continuously operating process that produces good quality PCC while the design parameters for larger scale were determined. The object of the phase 2 was to continue the work on the process design and cost estimate of the pilot-scale test facility (reactor size order 0.5 m3). In addition, construction was set to begin during phase 2.

In this report, construction process, and experience gathered from it, is shortly presented. In addition, first test drives and obtained results are described.

2 Construction of the pilot-scale test facility

Although the original plan was that the reactors are in size of 500 l, it was decided during the designing phase that 200 l reactors are better suitable for the plant. The pilot-scale design was completed in 2012, followed by ordering of the equipment, e.g. reactors, pumps, filters, sensors, etc. The construction work began in January 2013 and continued until the end of January 2014. The process flow diagram is presented in Figure 1.

2.1 Process design

This Slag2PCC concept consists of two stages; namely ***extraction stage*** where calcium is extracted from the steelmaking slag by using aqueous solutions of ammonium salt, and ***carbonation stage*** where the resulting calcium rich solution is reacted with a simulated flue gas (i.e. CO_2 and N_2) producing precipitated calcium carbonate (PCC).

In the extraction stage, calcium is extracted from the steel slag (0 -1mm) by using aqueous solution of ammonium salt under intensive mechanical mixing (reactor 1). The slag is transported to the reactor via a feeder (Sulkusyötin). After the set time (such as 30 minutes) the calcium–rich solution is separated from the residual slag via bag filter housing. The extraction reactor can currently be operated in batch mode, but later also in continuous mode.

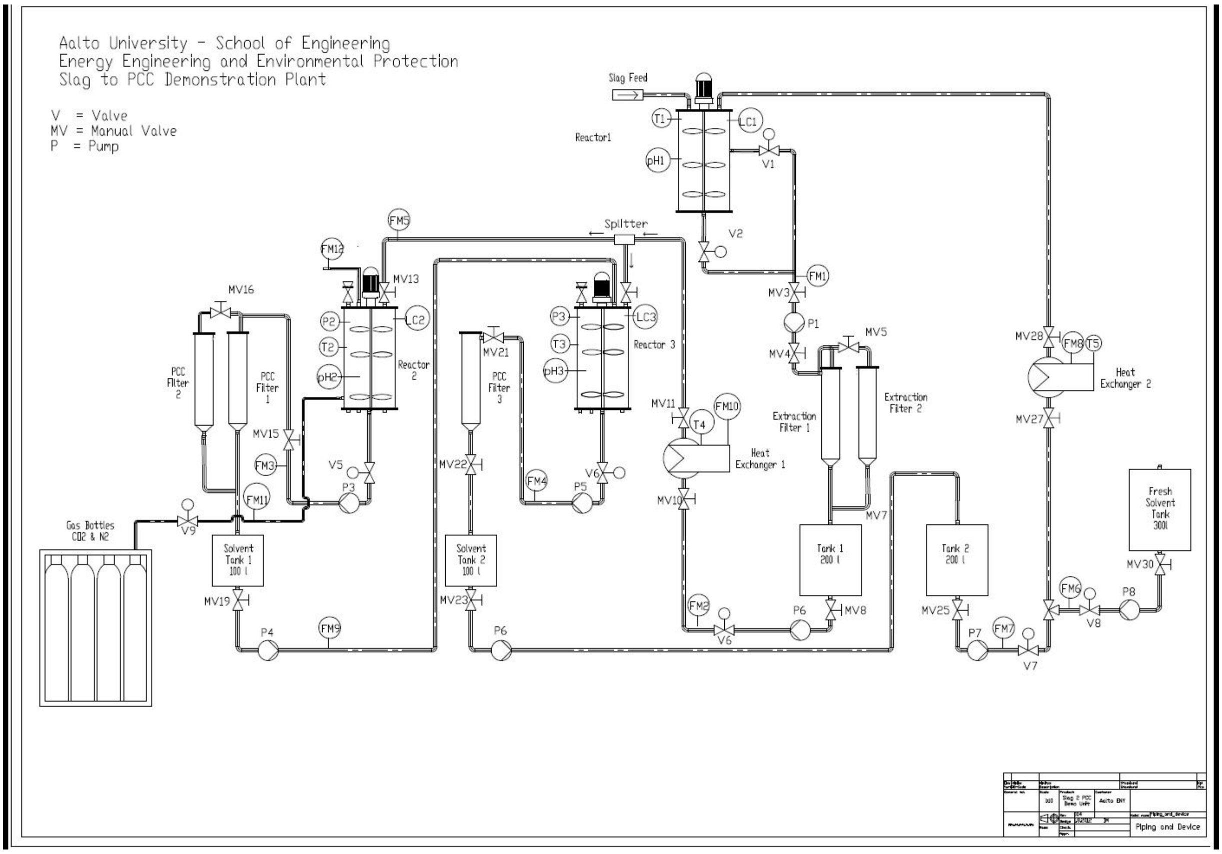

Figure 1. Process flow diagram

The calcium rich solution is split into the primary carbonation reactor (reactor 2) and secondary carbonation reactor (reactor 3). In the primary carbonation reactor, 90% of the calcium-rich solution is reacted with the carbon dioxide bubbled from the bottom of the reactor to form precipitated calcium carbonate (PCC). After filtration, the unreacted carbonate ions in the filtrate solution will react with the remaining 10 % of the original calcium-rich solution in the secondary carbonation reactor producing additional PCC product and purified ammonium salt solution to be reused for the extraction of calcium from slag in reactor 1.

2.2 Components of the pilot plant

The installation frame is an equilateral cube-shaped: 1800 mm x 1800 mmx 1800 mm standing steel frame of 3 floors (Figure 2). All process equipment is placed inside this frame. The plant consists of 3 (200 l) reactors, 5 bag filter housings, 2 heat exchangers, 5 reserve tanks, slag feeder, 8 pumps, 3 mixers, houses, pipes, pH and temperature sensors, and liquid and gas flow meters (Table 1). One of the challenges was that all the equipment in the markets are either designed for the laboratory level or for the industrial level, thus it was difficult to find suitable equipment for the test facility.

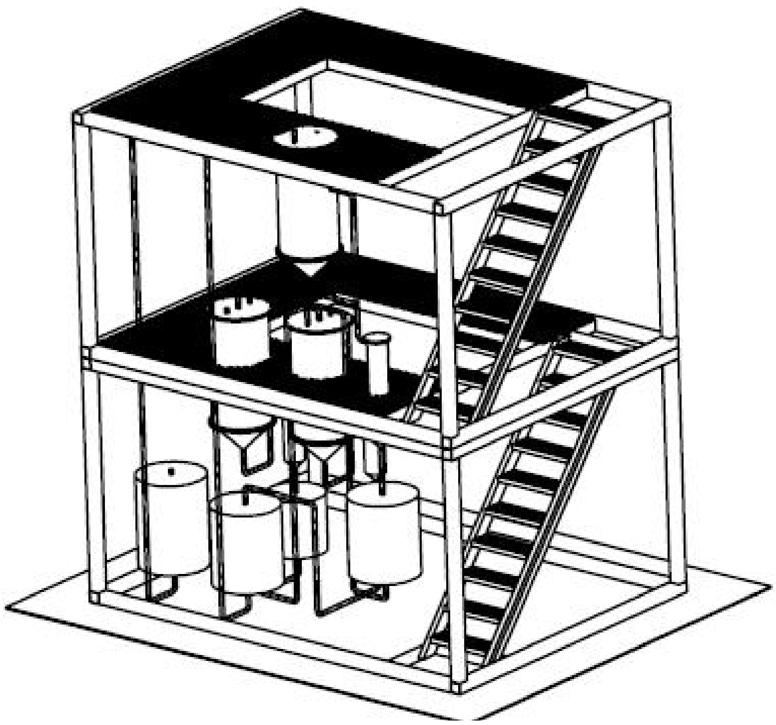

Figure 2. Installation frame

Table 1 Key components of the pilot plant

Name	Unit	Description	Index
Reactor 1(Extraction reactor)	1	Stainless steel AISI316 V = 200L, h = 1m, d = 50cm	h = hight d = diameter
Reactor 2 (Primary carbonation reactor)	1	Stainless steel AISI316 V = 200L, h = 1m, d = 50cm integrated with	
Reactor 3(Secondary carbonation reactor)	1	Stainless steel AISI316 V = 200L, h = 1m, d = 50cm integrated with	
Bag filter housing	5	ECS-02, Stainless steel AISI316	
Heat exchange	2	FP 40-59-1-NH Fixed plate	
Reserve tanks	5	2 x 200 L, 1 x 300L, 2 x 100L	
Slag feeder	1	RV-RVR 02, 2.2 l/r ,10rpm/min 0.37 kW	
Pump	8	SEF, Mag 22T8 0.25kW, 21l/min	
Mixer	3	CML 0.37 Kw, 170rpm HLS, 0.37kW 202RPM	
Hous		VEPA, 19 X 27MM EPDM 110C, 15 bar 3/4"	
Pipes		Stainless steel AISI316 3/4"	
pH sensor	3	Jumo , 0030u151 0 -12pH	
Temperature sensor	5	pt- 100	
Liquid flow meters	10	210, DN10 Max 32L/min	
Gas flow meters	2	Aalberg, GFM57 Max 10 kg/h	

2.3 Process instrumentation

The plant will operated and controlled automatically. In the event of any control system disturbances the plant can also be operated manually. All necessary process instrumentations and control system are now in place. Figure 3 illustrates an overview of the process control system.

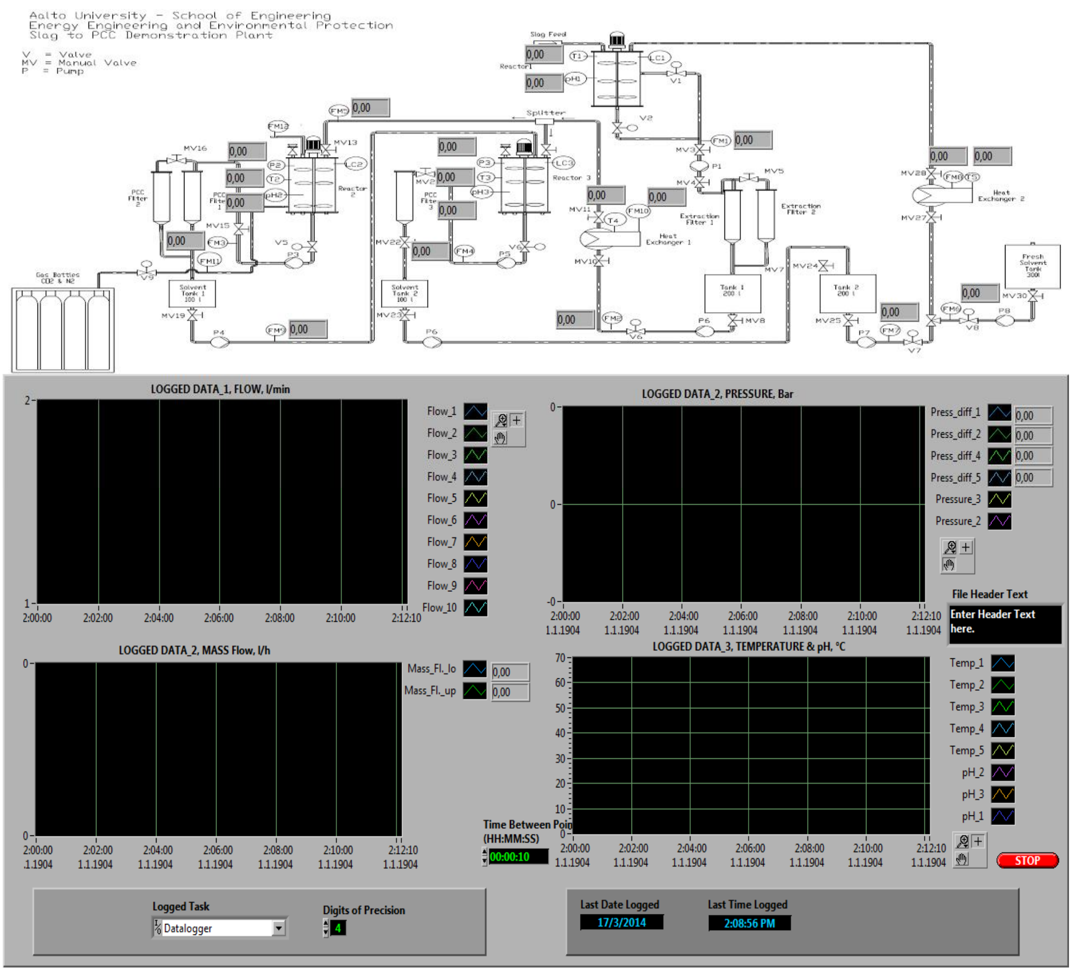

Figure 3. Process control system

3 First tests

The first set-up of the pilot-scale test facility was ready in January 27th in 2014. Next, a series of simplified tests using only water (no solids) was conducted. This showed some small leaks that were repaired shortly after the tests. The following test was done with water and steel slag. It was observed that the filter bag of 1µm was not enough; it caused the significant amount of particles below 1 µm to go through. Therefore a qualitatively filter of 0.45 µm was introduced which resulted with very clean filtrate solution.

After these simplified tests with water and slag, two “real” tests with 150 l of 1 M ammonium chloride (NH_4Cl) and 10 kg of slag in each test was conducted. After filtration, the filtrate was pumped to a reserve tank and carbonated the next day. The precipitation process was successful, however the color of the PCC produced from the two tests was not bright as it was expected. The color was a bit yellow, and thus investigations concerning this were started. Even though the gas distribution frame was supposed to be resistant to acid, we found that the welding was totally in rust, very likely causing the coloring of the precipitate. New gas distribution frame is supposed to arrive at any day now, after which the real experimental work can start.

During the extraction and carbonation tests, pH and temperature of the processes were measured; but, unfortunately due to some problems on the data logger the process data was not retrieved. Immediately after the tests were completed, the data logger problems were repaired, thus all the process data from the future tests should be available.

4 Conclusions

The object of the phase 2 was to continue the work on the process design and cost estimate of the pilot-scale test facility. In addition, construction was set to begin during the phase 2.

The pilot-scale design was completed in 2012, followed by ordering of the equipment. One of the challenges was that all the equipment in the markets are either designed for the laboratory level or for the industrial level, thus it was difficult to find suitable equipment for the test facility. However, the construction work began in January 2013.

The installation frame is an equilateral cube-shaped steel frame of 3 floors and all of the process equipment is placed inside this frame. The plant consists of (200 l) reactors, bag filter housings, heat exchangers, reserve tanks, slag feeder, pumps, mixers, houses, pipes, pH and temperature sensors, and liquid and gas flow meters.

The first set-up of the pilot-scale test facility was ready in January 27th in 2014. Next, a series of simplified tests using only water (no solids) were conducted. After the found leaks were repaired, the test was done with water and steel slag. This revealed that the set filters were not enough, thus a qualitatively filter of 0.45 µm was added resulting with very clean filtrate solution. These tests and repairs were followed with “real” tests using aqueous solution of ammonium salt and steel converter slag. The produced precipitate was slightly yellow. The gas distribution frame was likely the cause of problem (welding was rusty), thus new one has been ordered and should arrive at any day. The data logger problems that occurred during the tests have been repaired. The real experimental work is going to start soon.