Filtration tests of slag2PCC slurries manufactured at the Aalto pilot

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Abstract

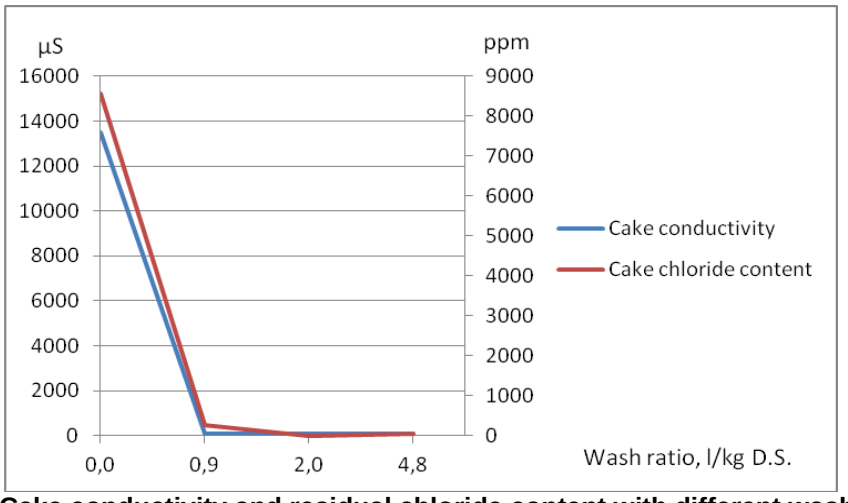
This report presents the results of filtration tests for the slag based precipitated calcium carbonate (slag2PCC) manufactured at the pilot plant of Aalto University. Two kinds of material were tested, one with the calcite type crystal structure, and one with the aragonite type structure. The tests were performed at Outotec Lappeenranta, where the materials arrived in a slurry form from Aalto. The lot sizes were about 10 kg of solids of both PCC types. The tests included pressure filtration and vacuum filtration, and the selected test units were Labox 100 and Büchner, respectively. The series contained altogether 21 different filtration cycles with varying combinations of filtering, washing, and drying steps. The filtered cakes were analyzed by their residual moisture content, chloride content and conductivity, and the filtrates by their residual solids content, chloride content and conductivity.

The objective of the test work was to determine the maximum filtration capacities of the PCC slurries, as a function of the thickness, moisture content and residual chloride content of the filtered cakes. The detailed information on the test procedure, and the results obtained are given in Appendix A. The results are summarized in the following text.

Both PCC slurries are easy to filter with both Outotec technologies, i.e. with pressure filtration and with vacuum filtration. Filter cloths AINO K11 (pressure filtration) and ARTO S11 (vacuum filtration) gave very clear filtrate for both slurries. The solids content of the filtrates were <10 mg/l and 10-15 mg/l, respectively.

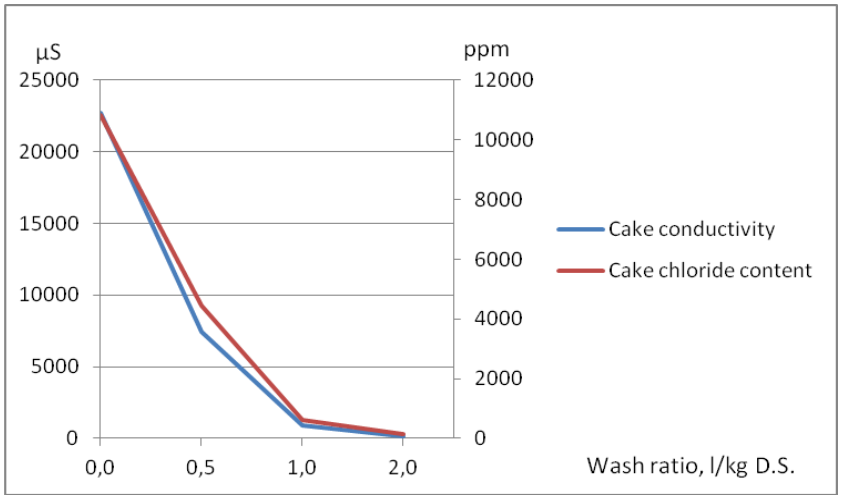
Calcite was filtered at room temperature and wash liquid temperature was also ~20 °C. Outotec Larox pressure filtration technology gave a high capacity and a low cake residual moisture content. Wash liquid penetrated the cake easily, even with a low wash water pressure. Washing can be performed either directly after the slurry feed, or after intermediate pressing. Compared to pressure filtration, vacuum filtration gave even higher capacities for the filtration of calcite slurry. On the other hand, the cake residual moisture stayed higher. Solids washing can be successfully performed also with Outotec vacuum filtration technology.

The cake residual chloride content and conductivity drop, when performing solids washing, are shown in the figure below. The chloride content of the calcite cake can be dropped from 10000 ppm to ~10 ppm with 2 l/kg D.S. wash ratio.



Cake conductivity and residual chloride content with different wash ratios of calcite slurry.

Aragonite slurry was heated to ~55 °C for the tests, which is the same as the production temperature of the material. Also for this slurry, high capacities were achieved with both filtration technologies. Pressure filtration gave lower cake residual moistures than vacuum filtration, but also lower filtration capacities. Different from the calcite slurry, room temperature water could not be used for solids washing because the wash liquid did not penetrate the cake. With 50°C wash liquid, washing could be performed successfully with both filtration technologies. Cake residual chloride content with pressure filtration technology was dropped from original ~5700 ppm to 109 ppm with 1.4 l/kg D.S. wash ratio. With vacuum filtration, the chloride content was dropped from >13 000 ppm to 127 ppm with 2.0 l/kg D.S. wash ratio. The cake residual chloride content and conductivity drop, as a function of solids washing are shown in the figure below.



Cake conductivity and residual chloride content with different wash ratios of aragonite slurry.

Further testing of both PCC grades is recommended for wash liquid consumption evaluation more accurately. Further tests for pressure filtration are recommended to be performed with a bigger test unit, such as PF 0.1. This is due to easier control of the wash liquid volumes used, because the wash liquid penetrates the cakes very easily.

The desired chloride content of the cake can be achieved with quite a small washing water amount, and the filtration rate is fast, around 350 kg/(m^2h) (dry solids). What is noted from the test work is that aragonite gave higher chloride concentration (around 100 ppm) in the cake than calcite (around 10 ppm). This is probably due to the particle size (P50 41 µm and P80 84 µm for calcite and P50 27 µm and P80 50 µm for aragonite) but also the crystal morphology might have some effect on the result. If the higher chloride content of aragonite cake is due to the particle size it means that a lot more efficient washing is needed if finer PCC is produced.

The equipment size for 20 t/h PCC production (160 000 t/a) would be a 57 m^2 filter, which is very small. A rough estimate for the total investment for that size of a filter is less than 1 million euros depending on the filter type. In this case it is desired to have a good washing result so the cheapest filter types are not an option.

Customer:	ORC
Contact Person(s):	Tuukka Kotiranta, Heljä Peltola
Country:	Finland
Place:	Pori
Application:	Slag PCC – Aragonite & calcite
Product of Test:	PCC
Case. No.:	10018731
Case Manager:	Heljä Peltola
Test Case No.:	10018731T1
Test Performed by:	Toni Auvinen
Date of Test:	1.-8.12.2014
Location of Test:	Lappeenranta
Test equipment:	Labox 100 & Büchner
Date of Test report:	20.3.2015
Test reported by:	Toni Auvinen

CONTENT

1. GENERAL INFORMATION

ORC has been in co-operation with VTT on PCC slurry filtration tests. Continuation of these tests was performed in Lappeenranta. VTT research report VTT-R-00372-14 results were used as a basis for this test campaign. The test material was produced at the pilot plant of Aalto University.

2. OBJECTIVE OF TESTS AND SELECTED TEST EQUIPMENT

Objective for the test work was to determine:
- cake thickness
- maximum filtration capacity
- moisture content of the cake
- cake handling
- washing of chlorides from the cake

Selected test units were Labox 100 (pressure filtration) and Büchner (vacuum filtration), because of the sample size and easy filterability of the slurry.

3. CUSTOMER PROCESS DATA AND SIMPLIFIED FLOWSHEET

NA.

4. PRODUCTION DATA REQUIREMENTS

Solids quantity: as high as possible

Filter cake:

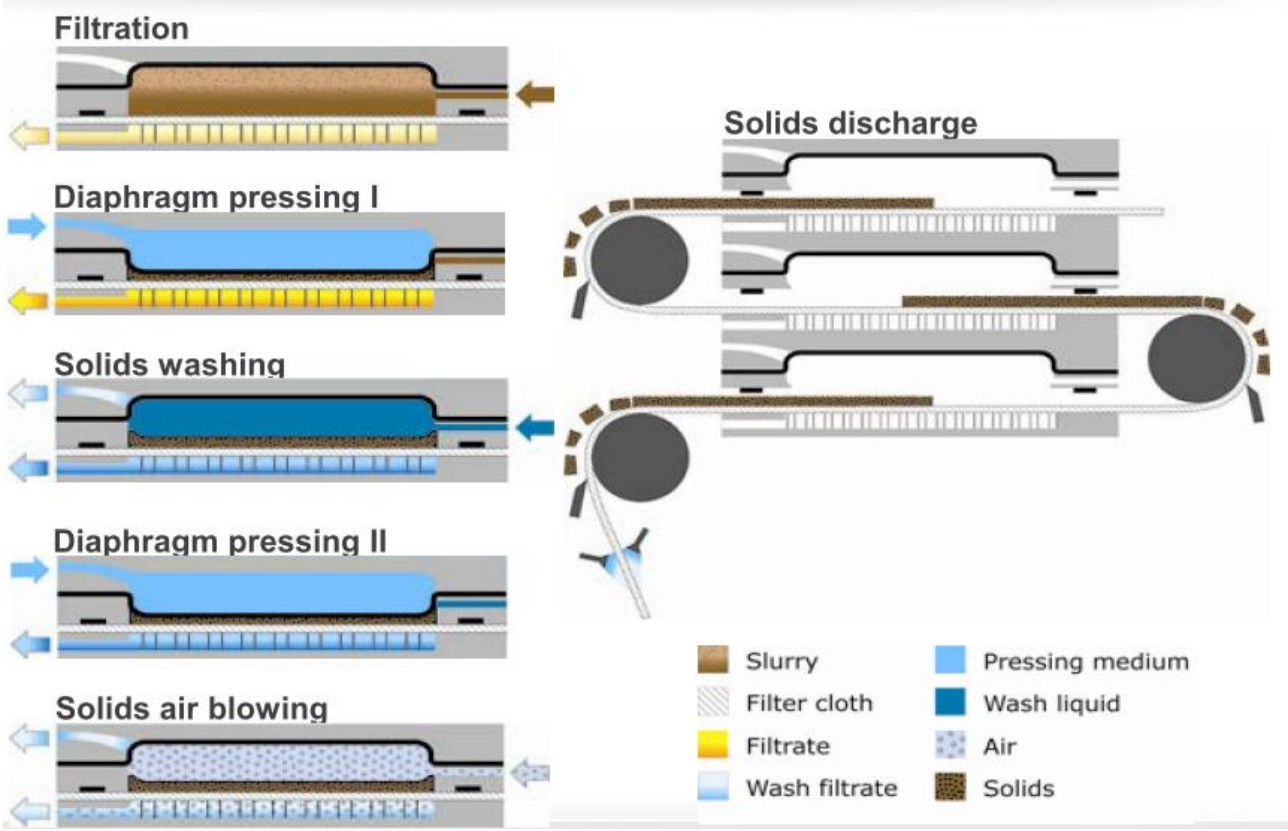
- cake moisture — as low as possible
- contains — minimum amount of chloride with reasonable wash liquid consumption

Filtrate:

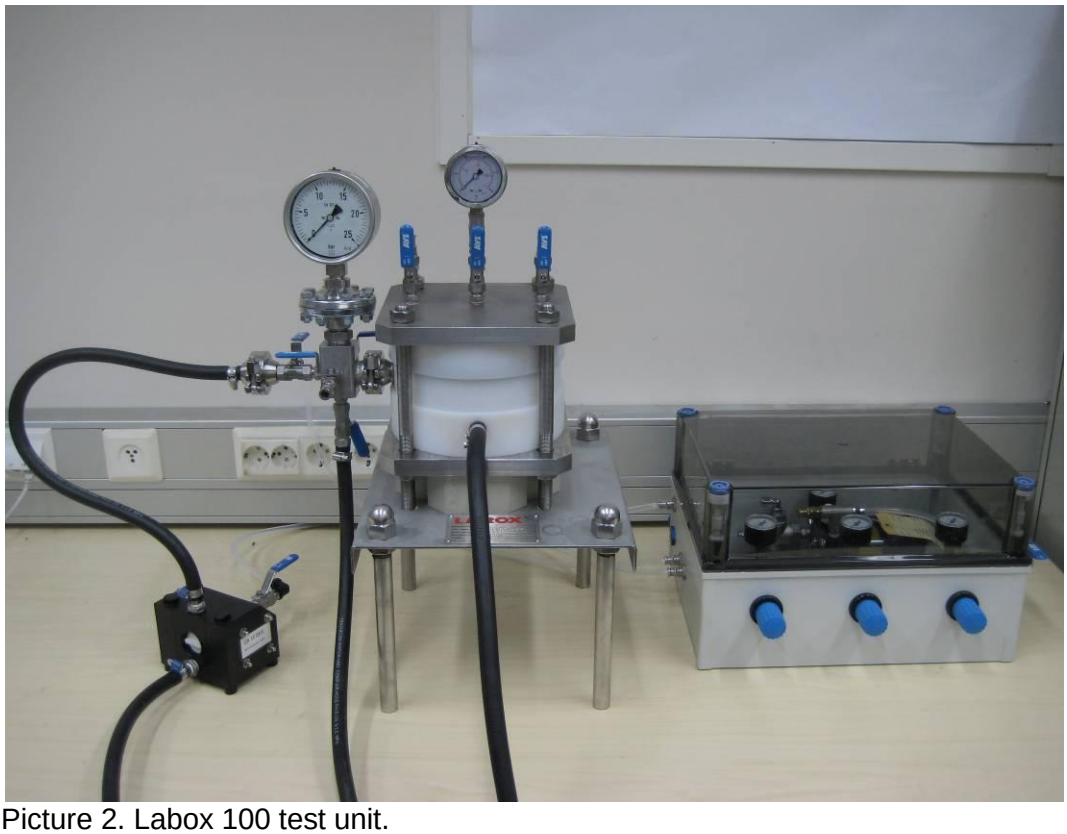
- clear filtrate

5. DESCRIPTION OF FILTRATION PROCESS AND SIMPLIFIED FLOWSHEET

In pressure filtration, the slurry is fed into the filter chamber by pumping. After pumping, the excess liquid is pressed out of the slurry inside the chamber (1st pressing). Pressure is produced by pumping water or air over a pressing diaphragm that expands and presses the slurry. After the 1st pressing, solids can be washed if needed. In solids washing, the wash liquid is fed into the empty space inside the chamber that has been created with 1st pressing. Then the wash liquid is pressed through the cake in 2nd pressing. After the cake has been pressed, the cake is air dried with pressurized air. Pressure filtration process is shown in picture 1. Labox 100 test unit is shown in picture 2.



Picture 1. Pressure filtration cycle.



Picture 2. Labox 100 test unit.

Outotec Larox® Büchner (BVB) test unit is used to simulate vacuum filtration. The test unit is used by pouring slurry sample into the cylinder, and then vacuum is applied to the slurry underneath the cake and the filter cloth. The time when there is excess water on the cake is called filtration time. When excess water has been sucked out of the cake, the vacuum is cut off, mother liquid is collected and then the cake can be washed. Washing is done by returning the vacuum on and by pouring the wash liquid on the cake with a spoon to ensure equal distribution of the washing liquid. When there is no more washing liquid on the cake surface, the cake drying begins. Washing can be performed in multiple stages and also in co-current or counter current mode. Büchner test unit is illustrated in picture 3.

Picture 3. Outotec Larox® Büchner (BVB) test unit.

6. TEST – PRODUCT AND WASH LIQUID DATA

Kind of process/product

- Product : Slag PCC
- Operation : Dewatering

Slurry	Calcite	Aragonite	
- Temperature	: amb	~55	^{o}C
- Density	: 1145	1307	g/l
- Solids content	: 24.8	40.2	wt%
- pH	: 7.3	7.2	
- Solids phase Composition	: PCC		
- Liquid phase Composition	: NH_4Cl		

Wash liquid

- Composition : Lappeenranta tap water
- pH : neutral
- Temperature : 24 50 ^{o}C

7. FILTRATION TEST DATA

The filtration test data of all the individual tests is given on the enclosure pages (p. 17-20).

Calcite

Outotec Larox PF (pressure filtration):

The following results are achieved with a chamber height of 60 mm by using AINO K11 filter cloth.

-	Specific filtration rate	398.8	kgDS/m^{2}h
-	Residual filter cake moisture	11.9	% w/w
-	Average drying air consumption	25	l/min
-	Solids in filtrate	<10	mg/l
-	Cake thickness	42	mm
-	Wash water consumption	1.8	l/kg DS
-	Wash result in cake	52	µS
-	Cake chloride content	10	ppm

The following filtration cycle parameters are illustrated in the test datasheet in test run #7 on page 17 (see enclosure).

-	Total cycle time	8.5	min
-	Pumping time in test unit	1	min
-	Cake washing	1.5	min
-	Pressing II	1	min
-	Air drying	1	min
-	Technical time	4	min
-	Pumping pressure	4	bar
-	Cake washing pressure	2	bar
-	Pressing pressure II	12	bar
-	Air drying pressure at start	3	bar
	after 1 min	3	bar

Outotec Larox RT / RB-SV / RT-GT (vacuum filtration):

The following results are achieved by using the filter cloth quality ARTO S11 filter cloth.

-	Test capacity	629	kgDS/m^{2}hr
-	Solid content in filter cake	78.9	%wt
-	Cake thickness	21	mm
-	Wash ratio	0.9	l/kgDS
-	Wash result	103	µS
-	Cake chloride content	252	ppm
-	Vacuum:		
	- Separation	0.5	bar
	- Washing	0.5	bar
	- Drying	0.4	bar
-	Air flow	15	l/min

The filtration parameters are illustrated in the test run #9 on page 18 of the attachment.

-	Separation	44	s
-	Intermediate drying	10	s
-	Washing	20	s
-	Drying	60	s

Aragonite

Outotec Larox PF (pressure filtration):

The following results are achieved with a chamber height of 60 mm by using AINO K11 filter cloth.

-	Specific filtration rate	453.1	kgDS/m^{2}h
-	Residual filter cake moisture	13.7	% w/w
-	Average drying air consumption	<10	l/min
-	Solids in filtrate	<10	mg/l
-	Cake thickness	52	mm
-	Wash water consumption	1.4	l/kg DS
-	Wash result in cake	110	µS
-	Cake chloride content	109	ppm

The following filtration cycle parameters are illustrated in the test datasheet in test run #15 on page 19 (see enclosure).

-	Total cycle time		9.5	min
-	Pumping time in test unit		1	min
-	Pressing I		0.5	min
-	Cake washing		1.5	min
-	Pressing II		1.5	min
-	Air drying		1	min
-	Technical time		4	min
-	Pumping pressure		4	bar
-	Pressing pressure I		8	bar
-	Cake washing pressure		6	bar
-	Pressing pressure II		12	bar
-	Air drying pressure	at start	6	bar
		after 1 min	6	bar

Outotec Larox RT / RB-SV / RT-GT (vacuum filtration):

The following results are achieved by using the filter cloth quality ARTO S11 filter cloth.

-	Test capacity	611	kgDS/m^{2}hr
-	Solid content in filter cake	74	%wt
-	Cake thickness	48	mm
-	Wash ratio	2	l/kgDS
-	Wash result	174	µS
-	Cake chloride content	127	ppm

- Vacuum:
  - Separation 0.5 bar
  - Washing 0.5 bar
  - Drying 0.5 bar
- Air flow 10 l/min

The filtration parameters are illustrated in the test run #21 on page 20 of the attachment.

- Separation 52 s
- Intermediate drying 10 s
- Washing 193 s
- Drying 60 s

8. ANALYSIS OF SAMPLES

Particle size distributions of the test materials (PCC calcite and PCC aragonite), measured by a laser particle size analyzer, are presented on the enclosure pages (p. 21-22).

The moisture contents of the filtered cakes were analyzed from approx. 50 g filtered samples (wet samples) which were dried in a laboratory oven at 60°C in air. The drying time was 38 h 15 min. The moisture contents were calculated from the mass losses of the samples. The results are given in Table A on the enclosure pages (p. 23).

Filtrate solids content was measured by pouring 100 ml of filtrate through filtrate paper. Paper with solids on was then dried in the oven overnight at 105 °C and weighed. Filtrate conductivities were measured with HANNA instruments conductivity meter. Cake conductivity was measured after mixing dry cake in ratio 1:1 with distilled water.

Cake chloride contents were measured by ORC. The chloride contents of the filtered cakes were determined as follows. A sample (approx. 10 g) was mixed with 100 ml (= 100 g) ion exchanged water. The suspension was stirred vigorously with a magnetic stirrer for 60 min. The suspension was filtered and the chloride content of the clear water solution was analyzed by an ion chromatograph (IC DX-120). In the case of the lowest chloride contents (cakes #6, #7, #10, #11), potentiometric titration with $AgNO_3$ was used instead of ion chromatography. In these analyses, increased sample weights (approx. 25 g) were also used.

The chloride contents corresponding to both wet cake and dry cake were calculated from the analyzed chloride content of the solution. The assumption for measuring the chloride contents by this manner was that all the chlorides in the filtered cake are water-soluble. It was also assumed that the chlorides were situated on the particle surfaces, and not locked inside the particles, so they were able to dissolve into water during stirring of the suspension. Two stirring times (30 min and 60 min) were tested before starting the analysis series, and they were found to give almost identical results. For the analysis series, the longer stirring time (60 min) was adopted. The analyzed chloride contents of the filtered cakes are presented in Table B on the enclosure pages (p. 24).

The chloride contents of the wash filtrates were analyzed by ion chromatography. The results are given in Table C on the enclosure pages (p. 25).

9. EXPLANATION OF TESTS

Slurry samples arrived in Lappeenranta in plastic containers. Slurry containers were not sealed properly and some of the liquid had been lost during transportation. Slurry was diluted when needed, with NH_4Cl liquid that was delivered with the slurry samples. Picture 4 shows the sample containers.

Picture 4. Container that had the slurry on the left, and container for the dilution liquid on the right.

Calcite

Test work began with calcite slurry. Slurry solids content was measured to be 60 % wt after some liquid had been lost during the transportation. Slurry was diluted with NH_4Cl solution to 24.8 % wt for the tests.

First tests were performed with Labox 100 pressure filter. Run #1 was performed with 33 mm chamber and with filter cloth AINO K11 that had been found to be the best in VTT research report. Slurry filtered very easily and chamber was full after ~40 seconds of pumping. Pumping was continued for 1.5 min. Pressing and drying were not performed due to a leakage in pressing air in the test unit caused by a mishap in the unit assembly. Cake solids content was above 70 % even without pressing and drying. Picture 5 shows the cake from test run #1.

Picture 5. Cake from test run #1.

Test run #2 was performed with 60 mm filter chamber as it was seen that slurry is easily filtered. Pumping pressure was also reduced to 4 bars for this reason. Chamber was full before 1 min slurry feed was performed. Pressing was then performed with 16 bar pressure for 1.5 min though after 30 seconds of pressing not much filtrate was collected. Pressing was followed by 1 min air drying with 4 bar pressure. Test resulted a hard cake with 12 % moisture content.

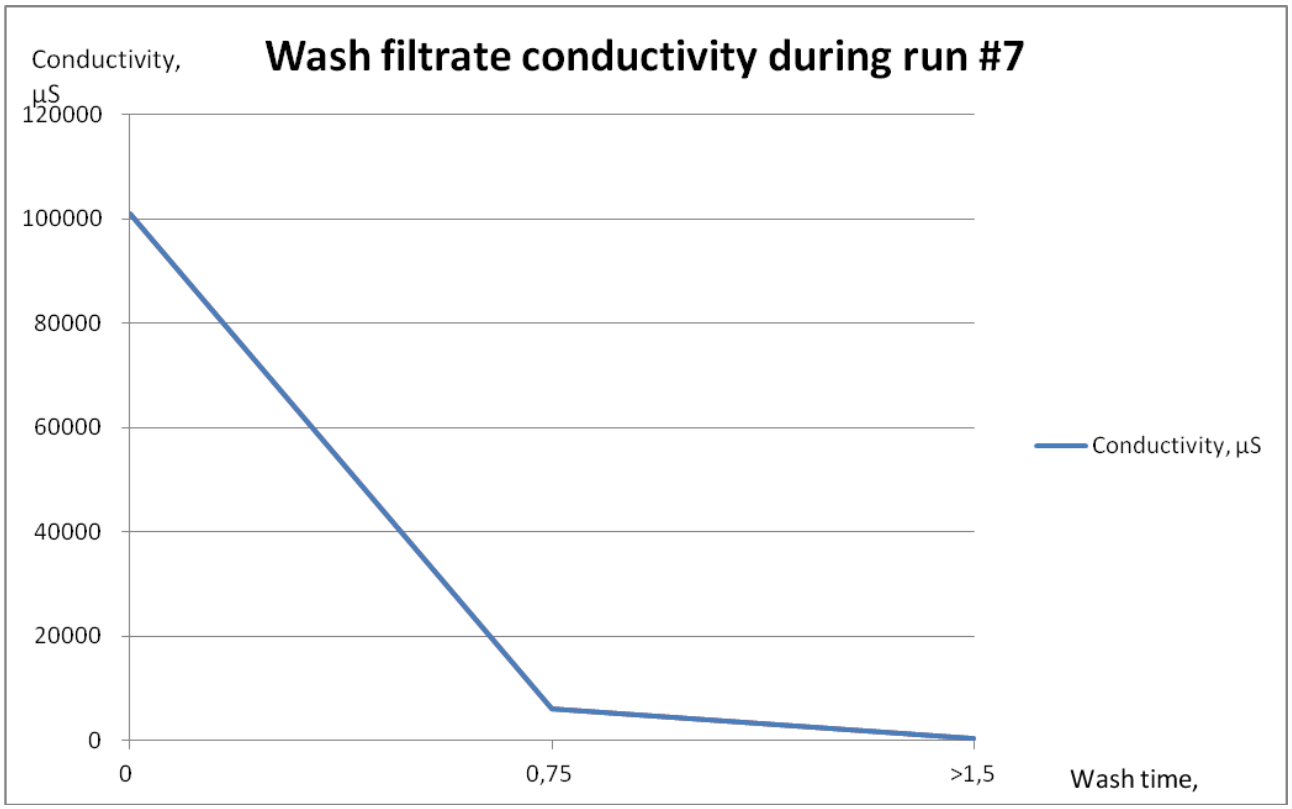
Büchner vacuum filtration tests were included as it was seen that the slurry is very easy to filter. Test runs #3-4 were performed with Büchner without solids washing. Filter cloth was ARTO S11 (tightest filter cloth for Büchner at the moment) and vacuum was 0.5 bar. Run #3 was performed with 500 ml slurry sample and run #4 with 1000 ml sample. This was done to optimize the filtration capacity. Bigger slurry sample gave much higher capacities and was therefore selected for further tests. Picture 6 shows the cake from test run #4.

Picture 6. Cake from test run #4.

Test runs from this point onward were performed with solids washing. Test runs #5-7 were performed again with Labox 100 pressure filter. Run #5 was performed with 1 min slurry feed, 0.5 min 1st pressing at 6 bar, solids washing of 0.5 min with 6 bar pressure, 1.5 min 2nd pressing and 1 min air drying. Wash liquid penetrated the cake very easily and in 30 seconds total of 1 liter wash liquid had been used. Wash filtrate conductivity was measured to be 1285 µS.

Test run #6 was performed with higher pressure in 1st pressing and lower wash liquid pressure. The 1st pressing was performed with 8 bar and washing with 2 bar pressure. Wash liquid pressure was dropped to 2 bars due to high wash liquid flow through the solids in previous run. Wash liquid volume of 1 liter was fed into the filter after 1 min 20 s.

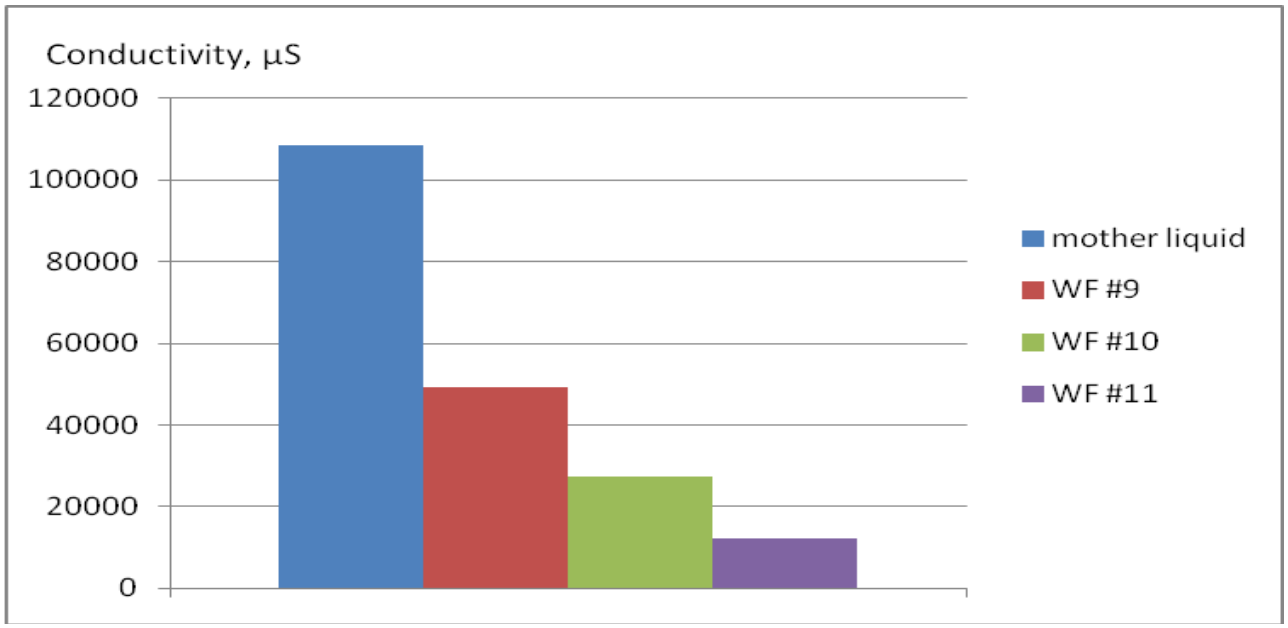
Test #7 was performed without 1st pressing. Wash liquid was introduced right after slurry feed. One liter of wash liquid was introduced to the filter in 1.5 min. Cake was then pressed and dried as in previous runs. Wash filtrate samples were taken at the beginning of washing, after 45 seconds of washing and during pressing. Picture 7 shows the conductivity measurements of these samples.



Picture 7. Wash filtrate conductivity during solids wash in run #7 (>1.5 min means filtrate from pressing).

Test runs #8-11 were performed with Büchner vacuum filter. Run #8 was performed for capacity evaluation as runs #3 and 4. Sample volume in run #8 was 750 ml. Results from runs #3, 4 and 8 show that the highest capacity was achieved with 1000 ml sample volume. This was therefore selected for washing tests.

Test runs #9-11 were performed with different wash ratios. Filtration cycle was otherwise the same in all 3 runs: after filtration, 10 s intermediate drying, solids washing and 60 s final drying. Wash liquid temperature was ~20 °C in all tests. Wash ratios were respectively 1, 2 and 5. All three cakes had residual moisture content of ~26 %. Cake conductivity dropped from original 13500 mS without washing to ~100 µS with 1 l/kg D.S. wash ratio. Higher wash ratios did not much affect the cake conductivity. Picture 8 shows how filtrate conductivity dropped in wash filtrate vs. mother liquid.



Picture 8. Conductivity of wash filtrates and mother liquid in test runs #9-11.

Aragonite

Tests with aragonite slurry were performed with slurry without dilution. Slurry density was 1.307 kg/dm^3 and solids content 40.2 % wt. Slurry was heated to ~55 °C and filter cloth in all tests was AINO K11. Slurry was first tested with Labox 100 pressure filter. Test run #12 was performed with 33 mm chamber without solids washing. Feed pressure was 6 bar and pressing pressure 16 bar. With 1 min feed, 1.5 min pressing and 1 min air drying, a 23 mm thick cake was achieved.

Test run #12 showed that aragonite slurry is about as easy to filter as calcite slurry. Further tests were therefore performed with higher 60 mm chamber. Pressures were also dropped in slurry feed to 4 bar and in last pressing to 12 bar. Test run #13 was performed without solids washing. Filtration cycle was the same as in run #12. Test resulted a 43 mm thick cake. Cake conductivity was 9100 µS.

Run #14 was performed with solids washing with room temperature water. Washing was performed right after slurry feed. It was seen that the wash liquid could not penetrate the cake. After 5 min of washing, only ~40 ml of wash liquid had been introduced to the cake though using 6 bar pressure.

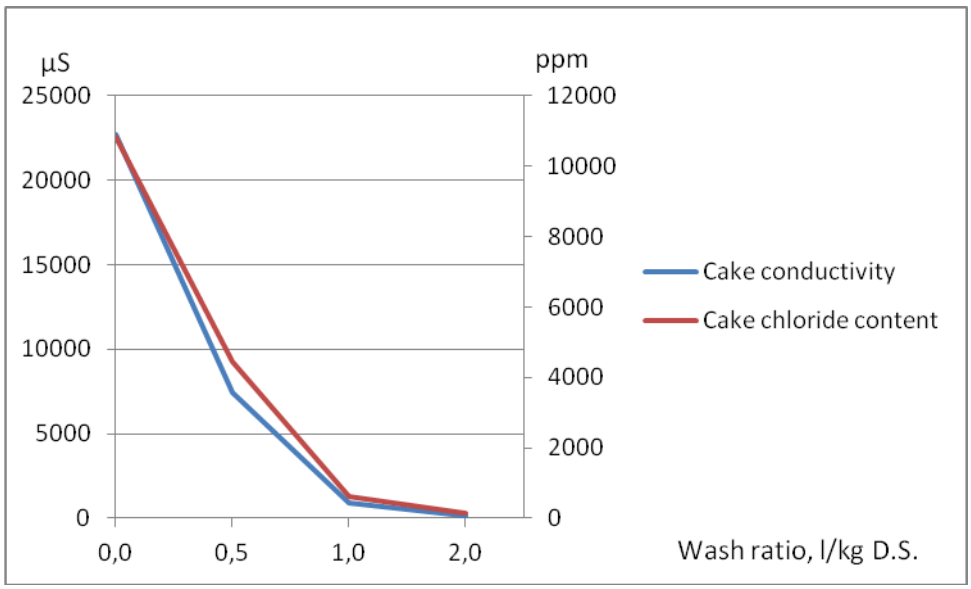
Run #15 was performed with solids washing after intermediate pressing. Wash liquid temperature was now ~50 °C. Slurry feed of 1 min was followed by 0.5 min intermediate pressing with 8 bar pressure. Then wash liquid was introduced to the cake with 6 bar pressure. After 1.5 min, 1 liter of wash liquid had been introduced to the cake. Cake thickness of 52 mm was achieved. Cake conductivity was measured to be 110 µS after washing. Cake from test run #15 is shown in picture 9.

Picture 9. Cake from test run #15.

Test runs #16-21 were performed with Büchner vacuum filter. Slurry temperature was again ~55 °C and filter cloth was ARTO S11. Runs #16-18 were performed with different sample volumes to find the maximum capacity. Slurry samples of 500, 750 and 1000 ml respectively were used. Cake thicknesses were respectively 24, 37 and 49 mm. Test run #18 with 1000 ml sample gave the highest capacity. Therefore that was selected for further tests with solids washing. Picture 10 shows the cake and mother liquid from test run #18.

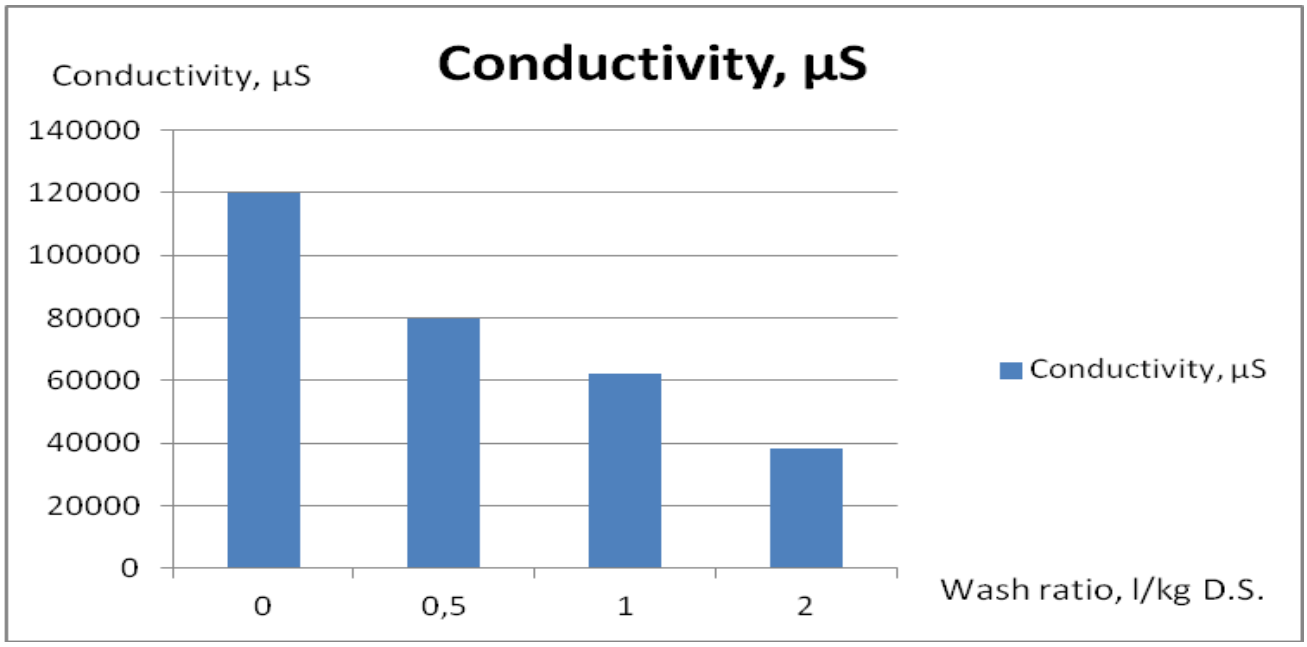
Picture 10. Cake and filtrate from test run #18.

Tests #19-21 were performed with solids washing. Wash water temperature was ~50 °C. Wash ratios of 0.5, 1 and 2 l/kg D.S. were used respectively. Before solids washing, separation phase was followed by 10 s intermediate drying. After washing, 60 s drying was performed. Picture 11 shows how the cake conductivity dropped without vs. with solids washing and how the cake residual chloride content varied between different wash ratios.



Picture 11. Cake conductivity and residual chloride content of aragonite with different wash ratios.

Picture 12 shows how the wash filtrate conductivities dropped with different wash ratios.



Picture 12. Filtrate conductivities with different wash ratios.

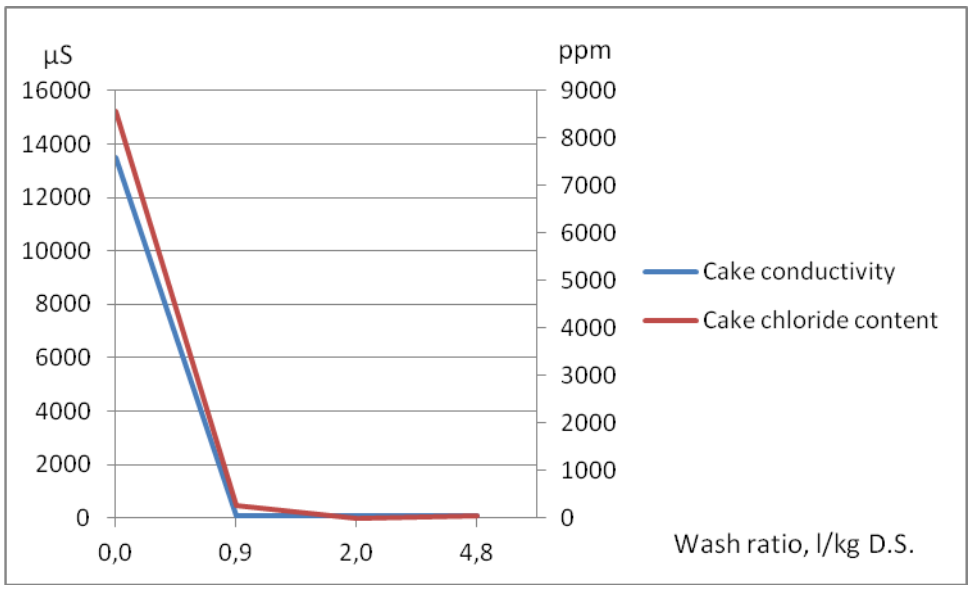
10. CONCLUSIONS AND RECOMMENDATIONS

Both PCC slurries are easy to filter with both Outotec technologies; pressure filtration and vacuum filtration. Filter cloths AINO K11 (PF) for pressure filtration and ARTO S11 (vacuum filtration) give very clear filtrate for both slurries. AINO K11 gave filtrate with <10 mg/l solids. With ARTO S11 the filtrate has slightly more solids in it, ~10-15 mg/l.

Calcite

Calcite was filtered at room temperature and wash liquid temperature was also ~20 °C. Outotec Larox pressure filtration technology gave high capacity and low cake residual moisture. Wash liquid penetrates the cake easily, even with low wash water pressure. Washing can be performed either directly after slurry feed, or after intermediate pressing. Chloride content of the cake can be dropped from 10000 to ~10 ppm with 2 l/kg D.S. wash ratio.

Vacuum filtration gives higher capacities for filtration of calcite slurry. On the other hand, the cake residual moisture stays higher with vacuum filtration. Solids washing can be successfully performed with Outotec vacuum filtration technology also. Cake residual chloride content and conductivity drop, when performing solids washing, are shown in picture 13.



Picture 13. Cake conductivity and residual chloride content of calcite with different wash ratios.

Aragonite

Aragonite slurry was heated to ~55 °C for the tests, which is the same as the production temperature of the material. For this slurry also high capacities were achieved with both pressure and vacuum filtration. Pressure filtration gives lower cake residual moistures but also lower capacities.

Room temperature water cannot be used for solids washing because the wash liquid does not penetrate the cake. With 50 °C wash liquid, washing can be successfully performed with both filtration technologies. Cake residual chloride content with pressure filtration technology was dropped from original ~5700 ppm to 109 ppm with 1.4 l/kg D.S. wash ratio. With vacuum filtration, the chloride content was dropped from >13 000 ppm to 127 ppm with 2.0 l/kg D.S. wash ratio.

Further testing is recommended for wash liquid consumption evaluation more accurately. Further tests for pressure filtration are recommended to be performed with a bigger test unit, such as PF 0.1. This is due to easier control of the wash liquid volumes used, because the wash liquid penetrates the cakes very easily.

Enclosures:
- Test datasheets (4 pages)
- PSD analyses (2 pages)
- Moisture contents of cakes. Chloride analyses of cakes and filtrates (3 pages)

Outotec

TEST FILTRATION DATASHEET **TEST FILTRATION NO.**

CUSTOMER	ORC	**DATE** 1.-2.12.2014	**ENCL. NO.**
BUSINESS UNIT		**BY** TonAuv	**PAGE NO.**
APPLICATION NAME	PCC - Calcite	**TEST PLACE** LPR	
SOLIDS DESCRIPTION		**WASH LIQUID TYPE** LPR tap water	
LIQUID DESCRIPTION		**PARTICLE SIZE DISTRIBUTION**	

Test unit type			**Labox100**	**Labox100**	**Labox100**	**Labox100**	**Labox100**					
Filtration area, m²			**0,01**	**0,01**	**0,01**	**0,01**	**0,01**					
Face area, m²			**0,01**	**0,01**	**0,01**	**0,01**	**0,01**					
Chamber depth, mm			**33**	**60**	**60**	**60**	**60**					
Test no			**1**	**2**	**5**	**6**	**7**					
Process conditions												
Density of	slurry	kg/dm³	1,145	1,145	1,145	1,145	1,145					
	liquid in slurry	kg/dm³	1,008	1,008	1,008	1,008	1,008					
S.G of solids in slurry	**calculated**	kg/dm³	2,572	3,094	2,947	2,512	2,529	0,000	0,000	0,000	0,000	0,000
Solids in slurry	**calculated**	%w/w	19,7	17,7	18,2	20,0	19,9	0,0	0,0	0,0	0,0	0,0
Density of	wash liquid	kg/dm³			1,000	1,000	1,000					
Temperature of	slurry	°C	17	17	18	18	18					
	wash liquid	°C			24	24	24					
pH of	slurry		7,3	7,3	7,3	7,3	7,3					
	wash liquid				7,0	7,0	7,0					
Filtration parameters												
Duration of	pumping	min	1,50	1,00	0,75	1,00	1,00					
	I pressing	min		1,50	0,50	0,50						
	washing	min			0,50	1,33	1,50					
	II pressing	min			1,50	1,00	1,00					
	drying	min		1,00	1,00	1,00	1,00					
	technical time	min	4,00	4,00	4,00	4,00	4,00					
Calculated cycle time		min	5,50	7,50	8,25	8,83	8,50	0,00	0,00	0,00	0,00	0,00
Measured process parameters during filtration tests												
Pressure of	slurry feed	bar	6,0	4,0	4,0	4,0	4,0					
	pressing I	bar		16,0	6,0	8,0						
	wash liquid	bar			5,0	2,0	2,0					
	pressing II	bar			16,0	12,0	12,0					
Quantity of slurry	**calculated**	l	1,8	2,7	2,0	2,3	2,5	0,0	0,0	0,0	0,0	0,0
Quantity of slurry	measured from tank	l										
Quantity of filtrate during	pumping	kg	1,51	2,12	1,68	2,04	2,03					
	pressing I	kg		0,12	0,017	0,01						
	washing	l			0,729	0,607	0,709					
	pressing II	l			0,185		0,106					
	air drying	kg		0,12	0,083		0,151					
	TOTAL w/o wash filtrate	kg	1,51	2,36	1,784	2,053	2,181	0	0	0	0	0
Consumption of	wash liquid	l			1,0	1,0	1,0					
Air flow / air pressure	beginning	l/min / bar		30/4	35/3	35/3	25/3					
	at 1 min	l/min / bar		30/4	35/3	35/3	25/3					
	end	l/min / bar										
pH of	filtrate											
	wash filtrate											
Process results												
Moisture in cake		% w/w	**29,3**	**21,3**	**19,4**	**12,0**	**11,9**					
Cake thickness average		mm	36	48	33	39	42					
Cake thickness variation		mm		46-49	32-34	38-40	40-42					
Wet cake weight		kg	0,587	0,693	0,524	0,608	0,642					
Dry cake weight	**calculated**	kg	0,42	0,55	0,42	0,54	0,56	0,00	0,00	0,00	0,00	0,00
We cake S.G.	**measured**	kg/dm³										
Wet cake S.G	**calculated**	kg/dm³	1,63	1,44	1,59	1,56	1,53	0,00	0,00	0,00	0,00	0,00
Filtration rate (dry solids)	**calculated**	kg/m²h	452,7	436,1	307,2	363,6	398,8	0,0	0,0	0,0	0,0	0,0
Filtration rate (filtrate)	**calculated**	l/m²h	1647	1888	1297	1395	1540	0	0	0	0	0
Wash liquid consumption	**calculated**	m³/ton D.S.	0,0	0,0	2,4	1,9	1,8	0,0	0,0	0,0	0,0	0,0
Wash liquid consumption (wash liquid/slurry)		vol / vol	0,0	0,0	0,5	0,4	0,4					
Washing result	cake conductivity	µS		7000,0	70,0	57,0	52,0					
Washing result	cake chloride content	ppm		10022,0		18,0	10,0					
Washing result	wash filtrate conductivity	µS			1285,0		330,0					
Solids ratio in cake (vol/vol)	**calculated**	%	44,8	36,7	43,4	54,6	53,2	0,0	0,0	0,0	0,0	0,0
Air in cake vs void volume	**calculated**	%	13,3	50,9	45,2	58,3	60,6	0,0	0,0	0,0	0,0	0,0
Solids content in	filtrate	mg/l		5								
Solids content in	wash filtrate	mg/l										
Filter cloth type			AINO K11	AINO K11	AINO K11	AINO K11	AINO K11					
Test date			1.12.	1.12.	2.12.	2.12.	2.12.					

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Buchner test filtration data sheet

BUCHNER: SEPARATION - 3 x WASHING - DRYING

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Company	**ORC**				Date						
Address					Tested by						
Reference					Page	1	of:				
Product	**PCC - Calcite**			Particle size	Wash liquids			Temp. °C		Test area [cm²]	
Grade					1st					standard	
Solids					2nd						
Liquid					3rd						

Test nr.				**3**	**4**	**8**	**9**	**10**	**11**				
Filtercloth				ARTO S11	ARTO S11	ARTO S11	ARTO S11	ARTO S11	ARTO S11				
dp wet filtercloth			bar	0,15	0,2	0,2	0,2	0,25	0,25				
Slurry	Temp.		°C	18	18	18	18	18	18				
	Amount		ml	500	1000	750	1000	1000	1000				
	Real d.s. content		g/l										
	Settling time		s	**5**	**5**	**5**	**5**	**5**	**5**				
	Vacuum		bar	0,5	0,5	0,5	0,5	0,5	0,5				
	Separation time		s	**15**	**44**	**28**	**44**	**50**	**52**				
	Drying time		s	**60**	**60**	**60**	**10**	**10**	**10**				
	Mother liquor		ml	420	840	625	800	785	780				
	Filtrate quality	solids	mg/l	15									
	Wash filtrate clarity												
Washing	1st	Temp.	°C				20	20	22				
		Volume in	ml				225	450	1125				
		Volume out	ml				250	480	1160				
		Wash time	s				**20**	**42**	**111**				
		Drying time	s										
	2nd	Temp.	°C										
		Volume (in/out)	ml										
		Wash time	s										
		Drying time	s										
	3rd	Temp.	°C										
		Volume (in/out)	ml										
		Wash time	s										
		Drying time	s										
Drying	Vibration		Yes/No										
	VSB time		s										
	Hot air/steam		°C										
	Air flow		l/min	15	15	15	15	15	15				
	Vacuum		bar	0,4	0,4	0,35	0,4	0,4	0,45				
	Drying time		s				**60**	**60**	**60**				
Cake	Thickness		mm	10	20	16	21	21	21				
	Cake cracking		Yes/No	no	no	no	no	no	no				
	Wet weight		g	139,98	301,6	237,95	307,45	305,92	304,99				
	Cake moisture		%wt	**21,5**	**24,7**	**23,69**	**21,1**	**25,04**	**23,66**				
	Dry weight		d.s. g	109,9	227,1	181,6	242,7	229,3	232,8				
	Cake conductivity		µS	13500			103	106	73				
	Cake chloride content		ppm	8553	8771	15133	252	3	48				
Total time			s	**80**	**109**	**93**	**139**	**167**	**238**				
Wash ratio			l/kg d.s.				**0,9**	**2,0**	**4,8**				
Test capacity			kg d.s./m² h	**494**	**750**	**703**	**629**	**494**	**352**				
Test capacity			l /m² h	**2250**	**3303**	**2903**	**2590**	**2156**	**1513**				
	Conductivities	mother liquid	µS	105000			108500	108400	108900				
		wash filtrate	µS				49100	27200	12300				
							WF #9	WF #10	WF #11				

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TEST FILTRATION DATASHEET

TEST FILTRATION NO.

CUSTOMER	ORC	**DATE**	4.-5.12.2014
BUSINESS UNIT		**BY**	TonAuv
APPLICATION NAME	Slag PCC - Aragonite	**TEST PLACE**	LPR
SOLIDS DESCRIPTION		**WASH LIQUID TYPE**	LPR tap water
LIQUID DESCRIPTION		**PARTICLE SIZE DISTRIBUTION**	

Test unit type			**Labox100**	**Labox100**	**Labox100**	**Labox100**							
Filtration area, m²			**0,01**	**0,01**	**0,01**	**0,01**							
Face area, m²			**0,01**	**0,01**	**0,01**	**0,01**							
Chamber depth, mm			**33**	**60**	**60**	**60**							
Test no			**12**	**13**	**14**	**15**							
Process conditions													
Density of	slurry	kg/dm³	1,307	1,307	1,307	1,307							
	liquid in slurry	kg/dm³	1,011	1,011	1,011	1,011							
S.G of solids in slurry	**calculated**	kg/dm³	2,550	2,483	2,193	2,570							
Solids in slurry	**calculated**	%w/w	37,5	38,2	42,0	37,3							
Density of	wash liquid	kg/dm³			1,000	1,000							
Temperature of	slurry	°C	55	53	55	53							
	wash liquid	°C			24	50							
pH of	slurry												
	wash liquid												
Filtration parameters													
Duration of	pumping	min	1,00	1,00	1,00	1,00							
	I pressing	min	1,50	1,50		0,50							
	washing	min			5,00	1,50							
	II pressing	min			1,50	1,50							
	drying	min	1,00	1,00	1,00	1,00							
	technical time	min	4,00	4,00	4,00	4,00							
Calculated cycle time		min	7,50	7,50	12,50	9,50							
Measured process parameters during filtration tests													
Pressure of	slurry feed	bar	6,0	4,0	4,0	4,0							
	pressing I	bar	16,0	12,0		8,0							
	wash liquid	bar			6,0	6,0							
	pressing II	bar			12,0	12,0							
Quantity of slurry	**calculated**	l	0,7	1,3	1,2	1,5							
Quantity of slurry	measured from tank	l											
Quantity of filtrate during	pumping	kg	0,276	0,605	0,663	0,79							
	pressing I	kg	0,108	0,136		0,078							
	washing	kg			0,037	0,97							
	pressing II	kg			0,137	0,111							
	air drying	kg	0,129	0,179	0,190	0,23							
	TOTAL w/o wash filtrate	kg	0,513	0,92	0,853	1,102							
Consumption of	wash liquid	l			0,040	1,0							
Air flow / air pressure	beginning	l/min / bar	10/6	10/6	<10/6	<10/6							
	at 1 min	l/min / bar	15/6	10/6	20/6	<10/6							
	end	l/min / bar											
pH of	filtrate												
	wash filtrate												
Process results													
Moisture in cake		% w/w	**11,7**	**13,5**	**11,0**	**13,7**							
Cake thickness average		mm	23	43	45	52							
Cake thickness variation		mm											
Wet cake weight		kg	0,383	0,735	0,771	0,85							
Dry cake weight	**calculated**	kg	0,34	0,64	0,69	0,73							
We cake S.G.	**measured**	kg/dm³											
Wet cake S.G	**calculated**	kg/dm³	1,67	1,71	1,71	1,63							
Filtration rate (dry solids)	**calculated**	kg/m²h	270,7	508,9	329,5	463,1							
Filtration rate (filtrate)	**calculated**	l/m²h	410	736	409	696							
Wash liquid consumption	**calculated**	m³/ton D.S.	0,0	0,0	0,1	1,4							
Wash liquid consumption (wash liquid/slurry)		vol / vol	0,0	0,0	0,0	0,7							
Washing result		%											
Cake chloride content		ppm	5657,0	5812,0	4655,0	109,0							
Washing result		%											
Solids ratio in cake (vol/vol)	**calculated**	%	57,7	59,6	69,6	54,9							
Air in cake vs void volume	**calculated**	%	53,5	42,6	37,8	49,7							
Solids content in	filtrate	mg/l	<10										
Solids content in	wash filtrate	mg/l											
Filter cloth type			AINO K11	AINO K11	AINO K11	AINO K11							
Cake conductivity		µS	9100	9700	8500	110							
Test date			4.12.	4.12.	4.12.	5.12.							

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Buchner test filtration data sheet

BUCHNER: SEPARATION - 3 x WASHING - DRYING

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Company	**ORC**				Date	**8.12.2014**					
Address					Tested by	**TonAuv**					
Reference					Page	1	of:				
Product	**Slag PCC - Aragonite**			Particle size	Wash liquids			Temp. °C	Test area [cm²]		
Grade					1st				standard		
Solids					2nd						
Liquid					3rd						

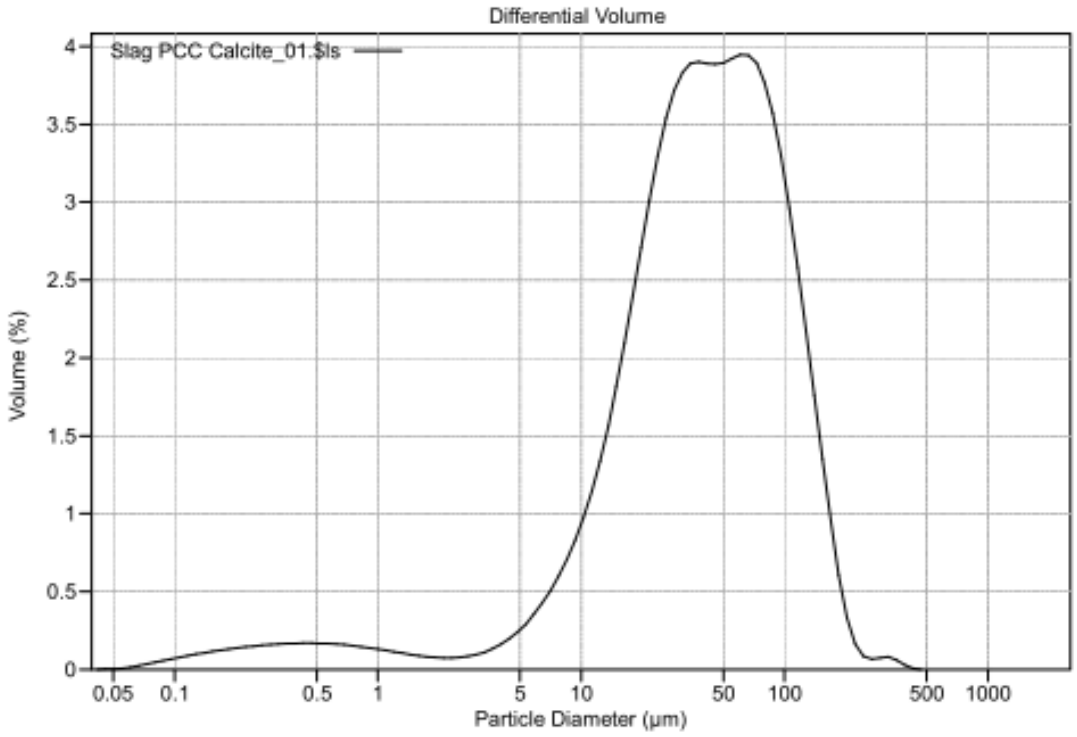
Test nr.				**16**	**17**	**18**	**19**	**20**	**21**			
Filtercloth				ARTO S11	ARTO S11	ARTO S11	ARTO S11	ARTO S11	ARTO S11			
dp wet filtercloth			bar	0,15	0,25	0,25	0,25	0,25	0,25			
Slurry	Temp.		°C	53	55	55	55	55	55			
	Amount		ml	500	750	1000	1000	1000	1000			
	Real d.s. content		g/l									
	Settling time		s	**5**	**5**	**5**	**5**	**5**	**5**			
	Vacuum		bar	0,5	0,5	0,5	0,5	0,5	0,5			
	Separation time		s	**15**	**31**	**52**	**61**	**54**	**52**			
	Drying time		s	**60**	**60**	**60**	**10**	**10**	**10**			
	Mother liquor		ml	290	440	580	530	515	520			
	Filtrate quality											
	Wash filtrate clarity											
Washing	1st	Temp.	°C				50	50	55			
		Volume in	ml				270	540	1080			
		Volume out	ml				340	590	1135			
		Wash time	s				**49**	**96**	**193**			
		Drying time	s									
	2nd	Temp.	°C									
		Volume (in/out)	ml									
		Wash time	s									
		Drying time	s									
	3rd	Temp.	°C									
		Volume (in/out)	ml									
		Wash time	s									
		Drying time	s									
Drying	Vibration		Yes/No									
	VSB time		s									
	Hot air/steam		°C									
	Air flow		l/min	15	10	10	10	10	10			
	Vacuum		bar	0,4	0,45	0,45	0,47	0,47	0,47			
	Drying time		s				**60**	**60**	**60**			
Cake	Thickness		mm	24	37	49	49	48	48			
	Cake cracking		Yes/No	no	no	no	no	no	no			
	Wet weight		g	357,6	544,3	742,1	754,3	730,3	734			
	Cake moisture		%wt	**25,6**	**25,99**	**27,96**	**25,6**	**28,38**	**26**			
	Dry weight		d.s. g	266,1	402,8	534,6	561,0	523,0	543,2			
	Cake conductivity		µS	21100	23000	22700	7400	895	174			
	Cake chloride content		ppm	13464	13993	10826	4455	598	127			
Total time			s	**80**	**96**	**117**	**185**	**225**	**320**			
Wash ratio			l/kg d.s.				**0,5**	**1,0**	**2,0**			
Test capacity			kg d.s./m² h	**1197**	**1511**	**1645**	**1092**	**837**	**611**			
Test capacity			l /m² h	**2250**	**2813**	**3077**	**1946**	**1600**	**1125**			

BECKMAN COULTER

LS Particle Size Analyzer

4 Dec 2014 10:46

File name:	C:\LS32\Samples\2014\Slag PCC Calcite_01.$ls Slag PCC Calcite_01.$ls
File ID:	Slag PCC Calcite
Sample ID:	000
Comment 1:	Calcite
Comment 2:	Analysed from dry sample
Optical model:	Fraunhofer.rfd PIDS included
Start time:	10:44 4 Dec 2014



Volume Statistics (Arithmetic) Slag PCC Calcite_01.$ls

Calculations from 0.040 µm to 2000 µm

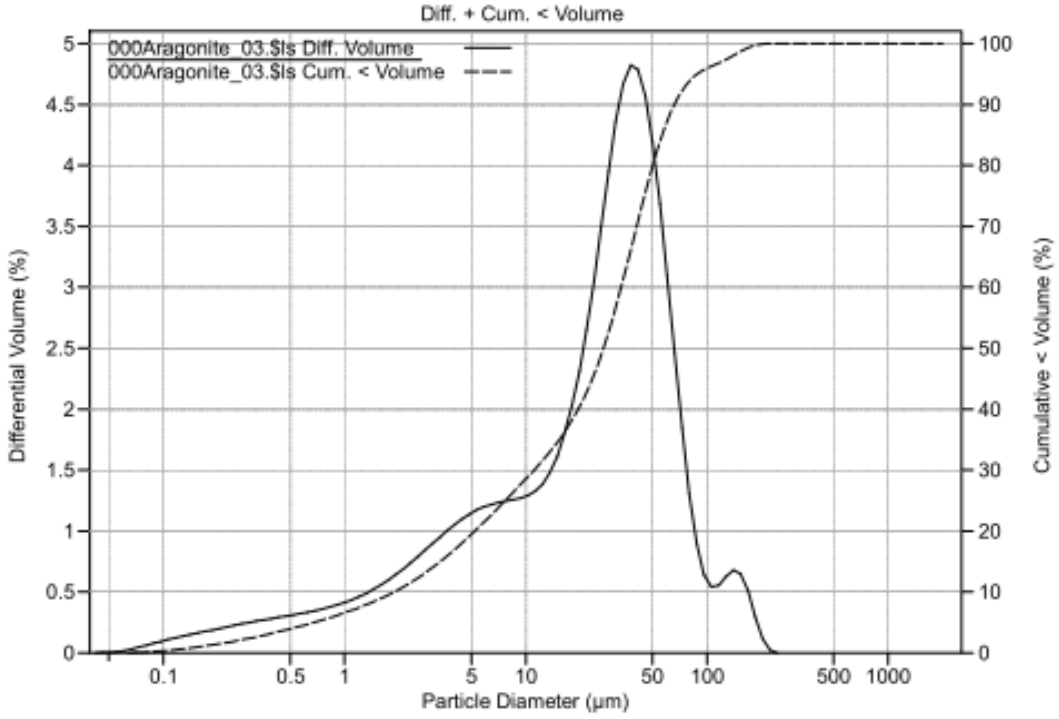
Volume:	100%		
Mean:	53.19 µm	S.D.:	44.05 µm
Median:	41.08 µm	Variance:	1940 µm^2
D(3,2):	5.175 µm	Skewness:	1.719 Right skewed
Mode:	60.52 µm	Kurtosis:	5.335 Leptokurtic

<10%	<20%	<50%	<80%	<90%
10.34 µm	18.30 µm	41.08 µm	84.26 µm	112.4 µm

BECKMAN COULTER

LS Particle Size Analyzer

23 Dec 2014 10:47



Volume Statistics (Arithmetic) 000Aragonite_03.$ls

Calculations from 0.040 µm to 2000 µm

Volume:	100%		
Mean:	32.40 µm	S.D.:	31.67 µm
Median:	26.89 µm	Variance:	1003 µm^2
D(3,2):	3.044 µm	Skewness:	1.966 Right skewed
Mode:	37.97 µm	Kurtosis:	5.498 Leptokurtic

<10%	<20%	<50%	<80%	<90%
1.869 µm	5.238 µm	26.89 µm	50.31 µm	66.31 µm

ANALYSES OF CAKES AND FILTRATES

Chloride contents of 19 filtered cakes and 23 filtrate solutions (wash filtrates) were analyzed at ORC. The moisture contents of the cakes were additionally analyzed.

Moisture contents

The moisture contents of the filtered cakes were analyzed from approx. 50 g filtered samples (wet samples) which were dried in a laboratory oven at 60°C in air. The drying time was 38 h 15 min. The moisture contents calculated from the mass losses of the samples are given in Table A.

Table A. Moisture contents of the filtered cakes.

Sample name	Wet sample	Dry sample	Weight loss	Moisture
	(g)	(g)	(g)	(wt.%)
PPC CAKE #2	51.34	40.39	10.95	21.33
PPC CAKE #3 DRIED	41.73	41.31	0.42	1.01
PPC CAKE #4 DRIED	50.15	49.79	0.36	0.72
PPC CAKE #6	51.10	44.97	6.13	12.00
PPC CAKE #7	52.06	45.85	6.21	11.93
PPC CAKE #8	50.27	38.36	11.91	23.69
PPC CAKE #9	49.60	39.16	10.44	21.05
PPC CAKE #10	50.23	37.65	12.58	25.04
PPC CAKE #11	51.73	39.49	12.24	23.66
PPC CAKE #12	50.51	44.62	5.89	11.66
PPC CAKE #13	49.08	42.48	6.60	13.45
PPC CAKE #14	51.16	45.55	5.61	10.97
PPC CAKE #15	54.58	47.08	7.50	13.74
PPC CAKE #16	50.39	37.49	12.90	25.60
PPC CAKE #17	52.49	38.85	13.64	25.99
PPC CAKE #18	61.05	43.98	17.07	27.96
PPC CAKE #19	50.31	37.42	12.89	25.62
PPC CAKE #20	51.41	36.82	14.59	28.38
PPC CAKE #21	50.19	37.14	13.05	26.00

Chloride analyses

The chloride contents of the filtered cakes were determined as follows. A sample (approx. 10 g) was mixed with 100 ml (= 100 g) ion exchanged water. The suspension was stirred vigorously with a magnetic stirrer for 60 min. The suspension was filtered and the chloride content of the clear water solution was analyzed by an ion chromatograph (IC DX-120). In the case of the lowest chloride contents (cakes #6, #7, #10, #11), potentiometric titration with $AgNO_3$ was used instead of ion chromatography. In these analyses, increased sample weights (approx. 25 g) were also used.

The chloride contents corresponding to both wet cake and dry cake were calculated from the analyzed chloride concent of the solution. The results are summarized in Table B. The assumption for measuring the chloride contents by this manner was that all the chlorides in the filtered cake are water-soluble. It was also assumed that the chlorides were situated on the particle surfaces, and not locked inside the particles, so they were able to dissolve into water during stirring of the suspension. Two stirring times (30 min and 60 min) were tested before starting the analysis series, and they were found to give almost identical results. For the analysis series, the longer stirring time (60 min) was adopted.

Table B. Chloride contents of the filtered cakes.

Sample name	Wet sample	Moisture	Chloride content, Cl^-				
			Analyzed from H_2O	Amount in 100 g H_2O	Chloride in wet cake	Chloride in dry cake	Chloride in dry cake
	(g)	(wt.%)	(mg/l)	(g)	(wt.%)	(wt.%)	(ppm)
PPC CAKE #2	10.2736	21.33	810	0.081	0.7884	1.0022	10022
PPC CAKE #3 DRIED	10.2162	1.01	865	0.087	0.8467	0.8553	8553
PPC CAKE #4 DRIED	11.5981	0.72	1010	0.101	0.8708	0.8771	8771
PPC CAKE #6	25.1712	12.00	4.0	0.0004	0.0016	0.0018	18
PPC CAKE #7	25.1886	11.93	2.2	0.0002	0.0009	0.0010	10
PPC CAKE #8	10.4784	23.69	1210	0.121	1.1548	1.5133	15133
PPC CAKE #9	10.0344	21.05	20	0.002	0.0199	0.0252	252
PPC CAKE #10	25.7072	25.04	0.6	0.0001	0.0002	0.0003	3
PPC CAKE #11	26.5793	23.66	9.8	0.0010	0.0037	0.0048	48
PPC CAKE #12	10.6054	11.66	530	0.053	0.4997	0.5657	5657
PPC CAKE #13	12.7236	13.45	640	0.064	0.5030	0.5812	5812
PPC CAKE #14	12.0636	10.97	500	0.050	0.4145	0.4655	4655
PPC CAKE #15	10.5986	13.74	10	0.001	0.0094	0.0109	109
PPC CAKE #16	10.4817	25.60	1050	0.105	1.0017	1.3464	13464
PPC CAKE #17	10.3793	25.99	1075	0.108	1.0357	1.3993	13993
PPC CAKE #18	12.1810	27.96	950	0.095	0.7799	1.0826	10826
PPC CAKE #19	12.3742	25.62	410	0.041	0.3313	0.4455	4455
PPC CAKE #20	12.8399	28.38	55	0.006	0.0428	0.0598	598
PPC CAKE #21	10.6810	26.00	10	0.001	0.0094	0.0127	127

The chloride contents of the wash filtrates were analyzed by ion chromatography. The results are in Table C.

Table C. Chloride contents of the wash filtrates.

Sample name	Chloride content
	(mg/l)
7.1 wash filtrate	35000
7.2 wash filtrate	2010
7.3 wash filtrate	40
CALCITE ML AINO KII #2	34000
CALCITE ML ARTO SII #3	35000
WF # 9	15000
WF # 10	8230
WF # 11	3500
WF # 14	33000
WF # 15 15.1	210
WF # 15 15.2	85
WF # 19	24000
WF # 20	17000
WF # 21	8800
ML # 9	35000
ML # 10	35000
ML # 11	35000
ML # 12	35000
ML # 14	36000
ML # 16	37000
ML # 19	39000
ML # 20	39000
ML # 21	40000