CCSP WP5.2.1, D542
2016

Hannu-Petteri Mattila
Ron Zevenhoven

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CLIC Innovation Oy
ETELÄRANTA 10
P.O. BOX 10
FI-00131 HELSINKI
FINLAND
www.clicinnovation.fi

ccsp
Carbon Capture and Storage Program

Åbo Akademi University
July 2016

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Hannu-Petteri Mattila*
Ron Zevenhoven

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FP4 report

* Currently at Paroc Group Oy, Parainen

Report Title: D542 – Solid-liquid separations in the Slag2PCC process

Keywords: solids separation, precipitated calcium carbonate, Slag2PCC, particle settling, hydrocyclone

Abstract

During FP 1 - 4 the Slag2PCC process concept has been scaled up from lab-scale experiments to a small demonstration scale plant, which now exists at Aalto University. Solid-liquid – separation processes for slag residue and calcium carbonate (PCC) product are essential regarding the overall economics of a possible industrial application in the future. The different solids in the aqueous process solutions present quite a few varying challenges and requirements for the separation equipment. While the slag particles used in most of the experiments had a median respective average diameters of 80 and 160 μm before calcium extraction, particle size of the produced PCC should be <2 μm, if it is to be used in commercial applications without further treatment. It is also possible to precipitate the carbonates as 20-50 μm particles that can be ground to smaller sizes. In addition, the slag residue contains small amounts of sub-micrometer size silica particles which need a distinct polishing filter before the carbonate precipitation step to prevent contamination of the solution in the carbonator and of the carbonates.

This report discusses the possibilities to use a so-called inclined settler or a hydrocyclone to pre-concentrate the slurries in the process before a filtration step, which would increase the time interval between successive filter shut-down and cleaning events, or the time between switching between two filter units. Both slag residue and PCC separation were studied. It seems to be very feasible to use a pre-concentrating device for concentrating the slurries, as a solids separation efficiency of 96 – 99 % is readily achieved, making the filtration step technically easier. For similar conditions otherwise, the inclined settler shows better performance than the hydrocyclone. The report ends with a few words on scale-up of the devices discussed, where again the inclined settler appears to offer better opportunities.This study is a continuation of the work reported as D512 (February, 2013).

Tuukka Kotiranta of Outotec, Pori, is acknowledged for comments (some as three footnotes in the text) and suggestions for the content of this report.

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Existing literature on particle separation in inclined settlers

Figure 1 shows the operational principle of an inclined settler (Davis and Gecol, 1996).

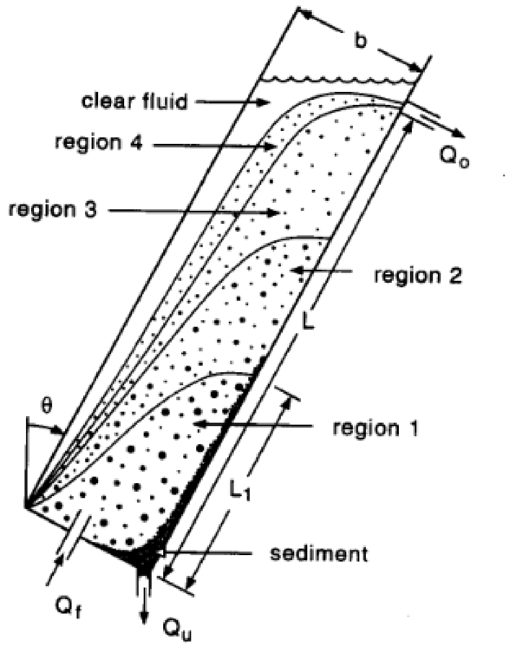


Figure 1. Schematic of inclined settler operation for four particulate phases (Davis and Gecol, 1996).

Besides the ÅA MSc thesis of Filppula (2012), available via the Clic CCSP project portal, and the literature sources mentioned therein, Thompson and Galvin (1997), for example, present a comprehensive summary of the essential features of inclined settlers. They studied a tube with an inner diameter of 25 mm and a length of 1.4 m, placed at an angle of 45°. The effects of particle size distribution, flow velocities, feed solids concentration and the ratio of particle settling velocity and flow velocity on the separation efficiency were presented. The authors conclude that an inclined settler provides a good cut-point control in size classification, being governed by the feed rate and the solids concentration. In their tests the d(50%) particle size, i.e. the size of which 50% of the particles were entrained in the overflow and 50% in the underflow, ranged from 2.6 to 116 µm.

Salem et al. (2011) present a CFD simulation of an inclined settler with three parallel channels. Due to a broad particle size distribution of the input material (crushed walnut shell particles in water, d(10%) = 35 µm, d(50%) = 115 µm, d(90%) = 235 µm) and other experimental parameters, the solid separation efficiencies were only 30-45%. More importantly, also the effect of different inflow configurations was studied, showing that this equipment feature can have up to 5%-points effect on separation efficiency.

Sarkar et al. (2007) performed a parameter study based on dimensional analysis, in more detail than Filppula. They included also the settling vessel dimensions to the analysis, an important factor not studied by Filppula (the number of required experiments otherwise being too large for an MSc thesis). Parameters such as settler angle, length and roughness of the settling plates (i.e. of the internal plates in the settler on which the particles “glide” downwards) as well as distance between them were identified as having importance. The flow properties were summarized with help of Reynolds and Froude numbers, i.e. based on the flow velocity and viscosity.

For the Slag2PCC application these results imply that it should be possible to concentrate the solid-containing slurries quite efficiently already with a simple inclined settler. Such a device could, with correctly tuned flow rates and other specifications also work as a size classifier, separating the larger agglomerates of >20 μm from the individual crystals in sizes of a few microns. [1]In both cases, the loading of the subsequent filters would be significantly reduced. Further development of the settler introduced and used by Filppula, for example by applying internal settling plates and optimizing the distances between those could further improve the unit performance.

Existing literature on particle separation in hydrocyclones

Hydrocyclones are seemingly simple devices that are widely used for separating liquids and solids, especially in the field of minerals processing. Still, most of the equations describing the performance of particle separation in hydrocyclones are based on empirical measurements and correlations between the hydrocyclone dimensions and the obtained results. For these reasons, the number of presented models is large.

As a basic model, e.g. Antunes and Medronho (1992) developed empirical correlations for two basic types of hydrocyclones (Table 1) as functions of Stokes (Stk) and Euler (Eu) numbers. Equations 1-7 and Table 2 summarize their findings. As can be observed, even the basic model consists of roughly twenty parameters, and cannot be seen as a fast approach to predict the practical performance of a hydrocyclone.

[1] For example, in a leaching process where the leachate residue contained significant amounts of very small particles the filtration could be improved by adding hydrocyclones to the thickener underflow (solid concentration ~ 400-700 g/l). The coarse particles were put to the belt filter and the fine particles were sent to the pressure filter that was better suited for the fine particles. The filtration capacity was thereby increased since both filters were operating at suitable particle size ranges. Also, washing efficiency of the leaching residue was improved which was the main target since the solution contained copper leached from the solids. Thus, there could be a market for inclined settlers if these can replace thickeners and hydrocyclones. However, the market is very conservative making it very hard to sell any innovative methods. Also, inclined settlers might not “pack” the underflow as much as settler does and as a result the solid concentration might be lower. In some applications that could be a problem.

Table 1. Families of geometrically similar hydrocyclones (Antunes and Medronho (1992)). D_i: inlet diameter, D_o: overflow diameter, D_c: hydrocyclone body diameter, l: length of vortex finder, L_i: length of the cylindrical part of the hydrocyclone, L: total cyclone length, θ: bottom angle of the cyclone.

Cyclone type	D_i/D_c	D_o/D_c	l/D_c	L_i/D_c	L/D_c	θ
Rietema	0.28	0.34	0.40	-	5.0	10-20°
Bradley	1/7	1/5	1/3	1/2	-	9°

$$Stk_{50} = \frac{(\rho_s - \rho) v_c d'^2_{50}}{18 \mu D_c} \tag{1}$$

$$Eu = \frac{\Delta P}{\frac{1}{2}\rho v_c^2} \tag{2}$$

$$Re = \frac{D_c v_c \rho}{\mu} \tag{3}$$

$$v_c = \frac{4Q}{\pi D_c^2} \tag{4}$$

$$Stk_{50} Eu = \alpha (\frac{ln}{R_w})^{\beta} e^{\gamma C_v} \tag{5}$$

$$Eu = \alpha_1 Re^{\beta_1} e^{\gamma_1 C_v} \tag{6}$$

$$R_f = \alpha_2 (\frac{D_u}{D_c})^{\beta_2} Eu^{\gamma_2} \tag{7a}$$

$$R_w = \alpha_2 (\frac{D_u}{D_c})^{\beta_2} Eu^{\gamma_2} \tag{7b}$$

Here: ρ_s: solids density, ρ: liquid density, v_c: superficial velocity, d_{50}: cut size, μ: liquid viscosity, ΔP: static pressure drop, Q: feed flow rate, n: parameter, R_f: Underflow-to-throughput flow ratio (Rietema), R_w: recovery of feed liquid to underflow (Bradley), C_v: feed solids concentration, D_u: underflow diameter

Table 2. Numerical values for parameters in equations 5-7 for families of geometrically similar hydrocyclones (Antunes and Medronho (1992)).

Cyclone type	α	β	γ	α1	β1	γ1	α2	β2	γ2
Rietema	0.0474	0.742	8.96	371.5	0.116	-2.12	1218	4.75	-0.30
Bradley	0.055	0.66	12	258	0.37	0	1.21E6	2.63	-1.12

Chen et al. (2000) evaluated the applicability of seven different hydrocyclone models for practical applications by running pilot tests with various liquid/solid combinations such as water/dust and organic solvent/salt. Their conclusion was that it is still necessary to calculate pressure drop over the entire cyclone, cut size (d50%), grade efficiencies (i.e. separation efficiencies of the different particle sizes) and the flow split (underflow/overflow rate) to be able to simulate the performance of a hydrocyclone.

Also, none of the tested seven models was able to describe all these parameters satisfactorily. According to Chen et al. it is of importance to apply the models only in the domains in which they were originally developed and tested.

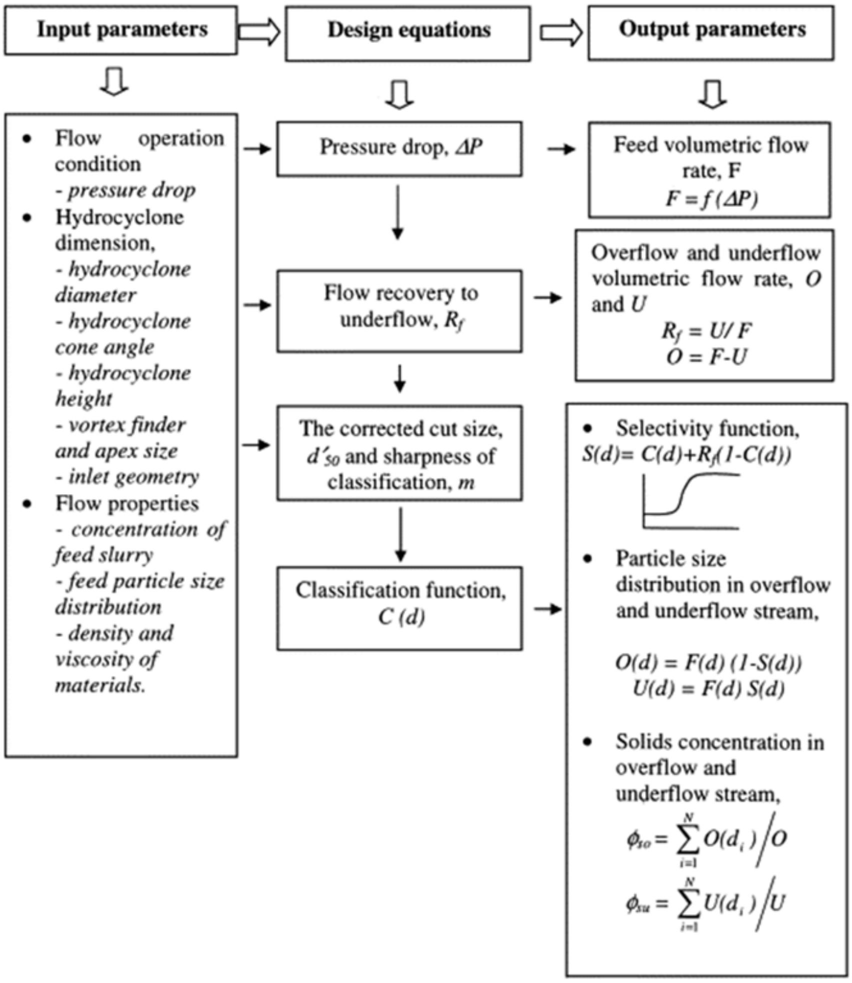


Figure 2. Hydrocyclone design procedure according to Kraipech et al. (2006).

A continuation of the work of Chen et al. was reported in 2006 by Kraipech et al. (Figure 2). The authors again noted that the two-phase flow does not allow only one set of equations to be used for properly describing all hydrocyclone systems. In addition, after “fine-tuning” several (eighteen individual equations) design equation sets by fitting the parameters to experimental data, they concluded that the design equations immediately require a re-fit if the hydrocyclone dimensions are changed. However, the models did well predict the consequences of changes in pressure drop or feed concentration without a parameter re-fit.

Narasimha et al. (2014) combined the empirical equation sets with CFD modeling, and studied a large number of experiments from the literature, in addition to experiments conducted by the authors. Their cyclone model seems to be able to include the changes in cyclone geometry and in the flow properties, giving a reasonable accuracy for the total flow rate, flow split to underflow and the reduced cut size. This is because the authors obtained certain additional and otherwise inaccessible parameters from the CFD model, including e.g. tangential velocities and hindered settling properties of the particles. Nevertheless, also this model is based on correlations between various dimensionless groups, and it should be pointed out that the authors used a wide range of feed solids concentrations in their model verification (3-70 %-wt). Especially the low end of the range is of interest for the Slag2PCC application.

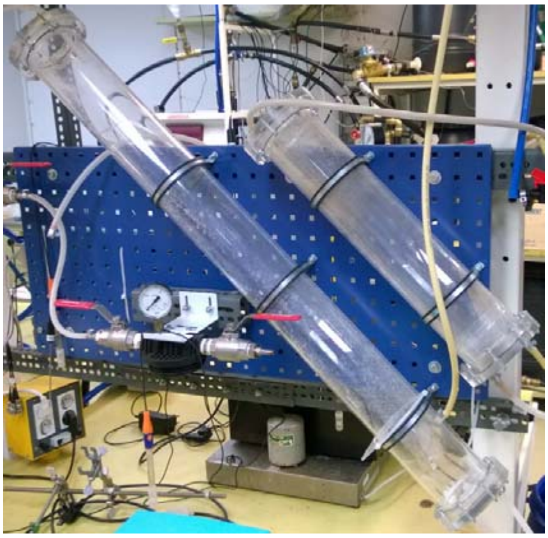
Roldán-Villasana et al. (1993) and Dueck et al. (2014) both focus on an interesting phenomenon which they call "the fish-hook effect". This involves the increased and somewhat unpredictable entrainment of the finest, near sub-micron range particles to the cyclone underflow, even if theoretically the smallest particles should exit the hydrocyclone in the ratio of liquid overflow/underflow. While the authors debate about the reasons for this phenomenon, it would in many cases be of importance to be able to predict the behavior of the fines. Also in the Slag2PCC process, both the silica-rich particles of the slag residue and the formed PCC are in this size category, and the efficiency of a hydrocyclone pre-separation unit would benefit from the additional separation of fines, thus reducing the load of the filter units. In fact, Dueck et al. (2014) provide equations to estimate the partition curve in the particle size range of the fish-hook effect while they also suggest that the phenomena could be avoided or controlled using multi-stage separation or by artificially changing the slurry PSD by adding e.g. coarse particles.

Probably the most relevant report is written by Neesse et al. (2015), discussing the use of a high pressure hydrocyclone for the sub-micron range particle classification. A 10 mm hydrocyclone was tested at up to 60 bar and up to 50 °C focusing on the submicron size particles. The fish-hook effect was clearly observed also in this study. As a conclusion the authors report an increase in the overall throughput of the cyclone, and a reduction in the particle cut size, down to 0.5 µm from a feed with maximum 10 µm particles. It appears to be still necessary to design a multistage separation procedure, and to accept an increase in the specific energy consumption in form of increased pump energy.

Separation of fine particles will require small (hydro-)cyclone devices, which for large process streams makes operation with a large number of cyclones necessary. It will be a challenge to properly distribute flows and avoid large differences in the separation efficiency of each cyclone.

Experimental work at ÅA

a)



b)

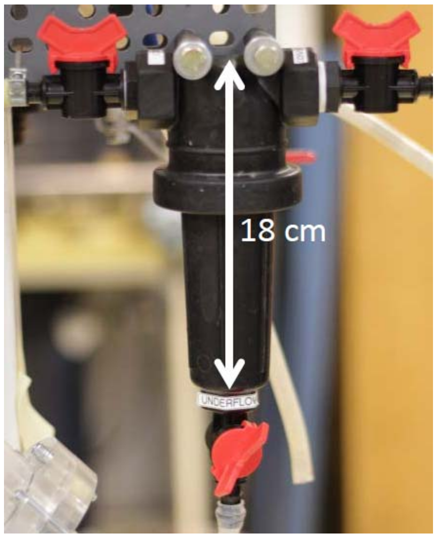


Figure 3. a) The inclined (45°) settler tubes used for experiments with PCC (left) and slag residue (right). b) A Miniclone of ChemIndustrial Systems, Inc.

Separation of slag residue and PCC particles was experimentally studied at ambient conditions with the tests listed in Table 3. Perspex tubes (Figure 3) with inner diameters of 10cm and lengths 50 and 110cm for slag residue and PCC, respectively, at an angle of 45°C were used as inclined settlers (Mattila and Zevenhoven, 2014). The experiments were initialized by filling the settlers with water.

A particle slurry, pre-mixed with a mechanical overhead stirrer (170 rpm), was then led into the settler. Both the overflow and underflow streams were mixed with the original slurry and recirculated as a feed stream for such a time (a few minutes) that, based on visual inspection, steady-state conditions were obtained in the settler. After that, a total slurry volume of 5 liters was led through the device with flow rates set so that a dilute overflow of roughly 0.5 L/min (30 L/h) was obtained.

For comparison, performance of a commercially available hydrocyclone (Figure 3) “Miniclone”, ChemIndustrial Systems, Inc. (Mattila, 2014), was also tested following a similar procedure.

The experimental results (Table 3) show that the tested equipment separates >96% of the solids both in case of the slag residue (different size fractions) and PCC. The hydrocyclone results in a smaller dilute outflow (40-50% of the inflow) than the inclined settlers (60-70%). It must be noted, however, that some particles remained inside the separation units during the tests, causing deviations in the overall mass balances.

Table 3. Separation experiments

Device	Solids	Size fract.	Flow rate, in	Inflow solids	Flow rate, out conc.	Outflow solids, conc.	Flow rate, out, dilute	Outflow solids, dilute	Separation efficiency
		µm	L/h	g/L	L/h	g/L	L/h	g/L	%
Inclined settler (50cm)	Slag residue	<38	51	36	16	115	35	0.48	99.1
Inclined settler (50cm)	Slag residue	125-250	49	30	19	75	30	0.54	98.9
Hydro-cyclone	Slag residue	53-75	50	87	29	128	21	8.20	96.1
Inclined settler (110cm)	$CaCO_3$	<10	50	37	15	87	35	0.05	99.9
Inclined settler (110cm)	$CaCO_3$	*	43	19	12	36	31	0.02	99.9
Hydro-cyclone	$CaCO_3$	<10	51	39	27	64	24	1.97	97.2

* PSD: <38 µm 15%, 38-53 µm 17%, 53-63 µm 15%, >63 µm 53%

Options for equipment scale-up

Scaling up the Slag2PCC process to industrial application in the end implies the production 20 t/h PCC. The currently existing demonstration unit at Aalto University in Espoo is capable of processing up to ~100 kg/h steelmaking slag, producing ~40 kg/h PCC. Yet the demonstration unit is only 6x larger than the set-up at ÅA and using an inclined separator as pre-separator also in Espoo would reduce the particle loading on the filters to (less than) 1% of what it is now – in practice it would mean that the time interval between filter cleaning increasing from (for example) 10 minutes to 1000 minutes, or once or twice per day.

Table 3 also shows the results for a hydrocyclone: with 96% efficiency for such a pre-separator the time interval between filter cleaning increasing from (for example) 10 minutes to 250 minutes, or once every four hours. [2]

[2] The solid concentration is typically only doubled for PCC and increased three times for slag residue. In many applications this is not enough. In conventional thickeners there are a few regions where solid concentration is increased. At top, the solids are just floating in water and settle to the lower part. At the bottom, a rake will make the slurry more thick and move it towards the output. At the output part the rake is designed so that it will further squeeze water from the wet solids. Thus, the inclined settlers ”replace” only (the first) part of the thickener. However, the design given in Figure 4 does not prevent adding a rake to the inclined settler to further increase the solid concentration. Basically, what is important is how much surface enables the settling and what kind of configuration gives a large settling surface area for equipment with a certain footprint. The conventional way is to increase the settler diameter (up to 100 meters or even bigger). The price for this kind of equipment comes from

Scale-up of an inclined settler from lab-scale as demonstrated at ÅA to the scale as used at Aalto requires only a factor of 6: this can readily achieved by putting six settlers as used at ÅA in parallel – see Figure 4 as an example (taking from fishery industry).

Furthermore, the report (Gonzalez et al., 1995) states for this type of settler: "*They are also commonly used to upgrade existing settling tanks since they have a higher separation rate*", apparently offering an alternative for more conventional cylindrical vessel settlers. Likewise, Thompson and Galvin (1997) mention inclined settlers as an alternative to hydrocyclones.

Either round tubes or a more compact honeycomb structure as shown in Figure 5 for seven tubes can be used. (For the latter a few tests are recommended to compare it to a round tube settler with the same hydraulic diameter.)

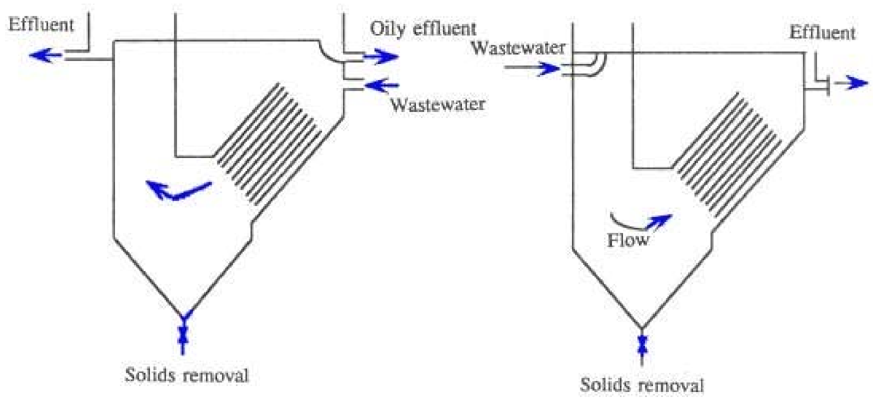


Figure 4. Multiple-channel inclined tube settler separation (Gonzalez, 1995)

Figure 5. Honeycomb structure arrangement for 7 parallel tubes

the large rake and the large vessel. In the inclined settlers the settling area could be increased while rake size would still be quite small. The drawback could be the maintenance: when some of the tubes start to block they may be very hard to access and open. Sometimes thickeners get blocked and emptying takes much time. With an inclined settler or hydrocyclone this may be more difficult and therefore it would not fit to just any kind of slurry.

Further scale-up towards 20 t/h PCC production implies another factor of 500 from the current demonstration scale at Aalto University. For this, a combination of conventional dimensional analysis (guaranteeing dynamic and geometric similarity – see Appendix) can be used to optimize the combination of an increased size of one settler unit and the number of units operated in parallel. Pumping power for transporting the liquid while achieving the required particulate pre-separation and separation cut size (d50%) that gives reasonable particle loading on the filters is the parameter to be optimized.

Scale-up of (hydro-)cyclones for applications where particles down to 1 μm or even below that are to be separated at a rate of several t/h is unattractive since it would require operation of a lot of small units in parallel, the dimensions of each dictated by the size of the particles to be separated. As mentioned earlier, distribution of flows and guaranteeing similar separation efficiencies for all units will be an additional challenge.

Besides this, another important feature points to a benefit of an inclined settler compared to a (hydro-)cyclone: in a cyclone, large particles may be taken up by the flow that eventually leaves via the top outlet, which should contain (if any) only the finest particles. It is much less likely that fines are taken out via the bottom outlet. For a settler the opposite holds: fines may be "trapped" by the bottom outflow while it is less likely that large particles leave via the top outflow. For the Slag2PCC process, in need of pre-separators upstream of barrier filters, the removal of large particles is more critical than the removal of fine particles.

Conclusions

- An inclined settler or a hydrocyclone can efficiently concentrate both the slag residue and the precipitated calcium carbonate from the process solution, thus reducing the particulate loading of the barrier filters.
- Particle retentions of >96% and >99% were achieved in the experiments with the hydrocyclone and inclined settler, respectively at the chosen conditions.
- The inclined settler gives a bottom slurry that is more concentrated than that of the hydrocyclone with otherwise similar flow conditions
- Use of this kind of devices would be advisable in the Slag2PCC process, because the barrier filters could then be dimensioned for smaller liquid flows, with significantly less filter shut-down and cleaning[3].
- Inclined settlers are readily scalable following scaling rules based on dimensional analysis; this is much less so for hydrocyclones.

[3] A for most research projects one conclusion here is: more research is needed. The solid concentration is very likely too small for industrial scale filters. A filter would have to be sized according to the slurry flowrate since so much solution must pass the filter. Normally a filter is sized according to the solid flowrate.

- It would be of interest to continue the experimental work with different particle size fractions and to compare the performance of the separation units to the calculated predictions from the several presented models, including also results from commercial software such as Aspen Plus.
- Especially, a comparison of theoretical and practical results of sub-micron scale particle separation would be an interesting part of future work.

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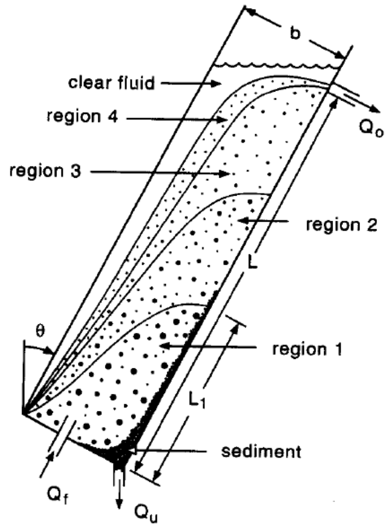
Appendix



Dimensional analysis allows for describing the performance of (in this appendix) an inclined settler as a function of several dimensionless groups. Scale-up can be based on dynamic similitude expressed as (dimensionless) force ratios and geometric similitude expressed by (dimensionless) size ratios.

As shown in the figure, flow Q_f (m^3/s) entering the separator contains a concentration c_{in} (kg/m^3) particles with (average) size d_p (m) and density ρ_p (kg/m^3) in liquid with density ρ (kg/m^3) and dynamic viscosity η (Pa·s). A liquid flow Q_o (m^3/s) leaves the separator as overflow, with particle concentration c_{out} (kg/m^3), while most of the particles leave the separator with underflow Q_u (m^3/s). The separator, a cylinder with diameter b (m) and length L (m) is inclined with angle θ (typical 45 – 60°) with respect to vertical. During operation, the particles form a downwards-moving layer of sediment with length L_1 (m) at the bottom.

A dimensional analysis of the efficiency, expressed as $c_{out}/c_{in} = f(Q_f, Q_o, d_p, \rho_p, \rho, \eta, L, b, L_1, g\cdot\sin(\theta))$, with gravity g gives eight dimensionless groups, since the eleven variables have three base units: kg, m and s. Note that the product g·sin(θ) can be taken as one variable (because g is a constant), and that Q (m^3/s) = ¼·π·b^2·v, with velocity v (m/s).

Using units to variables conversion $m \to L,\ \ kg \to L^3 \cdot \rho,\ \ s \to L^3 / Q_f,\ \ gives:$

$$\frac{c_{out}}{c_{in}} = f\left(\frac{Q_f}{Q_f}, \frac{Q_o}{Q_f}, \frac{d_p}{L}, \frac{\rho_p}{L^3 \cdot \rho \cdot L^{-3}}, \frac{\rho}{L^3 \cdot \rho \cdot L^{-3}}, \frac{\eta}{L^3 \cdot \rho \cdot L^{-1} \cdot Q_f \cdot L^{-3}}, \frac{L}{L}, \frac{b}{L}, \frac{L_1}{L}, \frac{g\sin\theta}{L \cdot Q_f^{\,2} \cdot L^{-6}}\right) =$$

$$f\left(\frac{Q_o}{Q_f}, \frac{d_p}{L}, \frac{\rho_p}{\rho}, \frac{\eta}{\rho \cdot Q_f \cdot L^{-1}}, \frac{b}{L}, \frac{L_1}{L}, \frac{g\sin\theta}{Q_f^{\,2} \cdot L^{-5}}\right),\ \text{where}\ \ Q_f = \frac{\pi}{4} b^2 v_f = \frac{\pi}{4} L^2 \frac{b^2}{L^2} v_f$$

$$\to \frac{Q_f}{L} \sim L v_f \to \frac{\eta}{\rho \frac{Q_f}{L}} \sim \frac{\eta}{\rho L v_f} = \frac{1}{\mathrm{Re}_f}\ \ \text{and}\ \ \frac{Q_f^{\,2}}{L^5} \sim \frac{v_f^{\,2}}{L} \to \frac{g\sin\theta}{Q_f^{\,2} \cdot L^{-5}} \sim \frac{gL\sin\theta}{v_f^{\,2}} = \frac{\sin\theta}{Fr_f}$$

$$thus: \frac{c_{out}}{c_{in}} = f\left(\frac{Q_o}{Q_f}, \frac{d_p}{L}, \frac{\rho_p}{\rho}, \mathrm{Re}_f, \frac{b}{L}, \frac{L_1}{L}, \frac{\sin\theta}{Fr_f}\right)$$

with Reynolds and Froude numbers Re and Fr. Scale-up should as much as possible satisfy that the dimensionless groups have the same value independent of size.