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Abstract: Various devices in liquid-solid separation require mechanical cake discharge using different technologies. An incomplete cake discharge causes yield losses, cycle time increase, irregular cake formation, high mechanical constraints which decrease medium life time, excess consumption of cleaning liquors….Currently, cake discharge is only assessed qualitatively by observations of filter cloth after discharge or quantitatively by weighing recovered solids. A prototype has been developed to carry out reliable and repeatable measurements by removing filter cloth from filter cake by shearing. This study presents results of experiments performed with calcium carbonate (which forms sticky cakes) and discusses the conditions of cake formation and cake detachment from filter cloth. It highlights the role of two main parameters : filter cloth characteristics and pressure.

Keywords: cake discharge; filtration cycle, filter cloth, adhesion strength, shearing, calcium carbonate

Introduction

In liquid solid separation, cake discharge contributes in the same manner as cake filtration, washing or deliquoring in the process cycle time. Various devices in liquid-solid separation (filter press, belt filter, leaf filter, candle filter, rotary drum filter, Nustche filter, filtering centrifuge…) require a mechanical cake discharge. Different techniques can be used: gravity discharge, vibrations, scraping, traction, shearing, centrifugation, chocks,… This key step is often underestimated in the process cycle time. An incomplete cake discharge causes yield losses, cycle time increase, irregular cake formation, high mechanical constraints. The choice of adapted operating conditions such as filter cloth characteristics, sludge pretreatment, cake thickness, pressure….is necessary to decrease discharge time to a minimum. Industrial feedback shows that cakes which are stuck to filter cloth and difficult to discharge, cause usually important productivity losses and require higher cleaning liquor volumes. Moreover, a poor discharge leads to a decrease of the filter cloth efficiency (which is a key point of filtration success) owing to phenomena as blinding, clogging (Weigert T. et al, 1996).

The parameter which may be associated to cake discharge is the adhesion force (or energy). It represents the mechanical force required to separate particles from a solid surface (Dutschk, 2000). In the field of liquid separation, cake detachment is usually assessed qualitatively by observing the cake fall from a vertical filter cloth and the remaining particles trapped at its surface. Only few researches were carried out to quantify adhesion forces between a filtration cake and a filter cloth and no devices are commercially available to carry out this measurement. Different methods were tested in the eighties to separate cake from filter cloth and to measure the corresponding forces by centrifugation (Muller et al., 1986, Salazar-Banda et al, 2007) or by shearing (Ward et al., 1971). The measurement of cake adhesion has been more specifically studied in other fields like in dust and gas filtration using air jet pulses, or in food products using impulse or centrifugal techniques for cake removal (Morris et al., 1991, Tanabe et al., 2011). In these cases, the measurements were conducted by combination of atomic force microscopy and angle contact measurement angle with a glass surface (Handojo et al., 2009, Shimada et al., 2002), by probe "Tack tests" for mortars (Kaci et al.,2011) or by different standardized methods for adhesives (Roche, 2011). These methods remain difficult to apply and other specific techniques have to be developed to assess the magnitude of the adhesion forces between filter cakes and filter clothes.

The study aims at studying the formation and discharge of a calcium carbonate cake from the filter cloth and its detachment from the filter cloth with a prototype recently developed to perform repeatable measurement of the adhesion force and energy between filter cake and filter cloth. The influence of filter cloth characteristics and pressure on cake detachment was studied.

Material and Methods

Cake formation

Cakes were formed by filtration of a 250 g/L calcium carbonate suspension on a filter cloth under pressure applied by a piston in a filtration-compression cell of 70 mm diameter. The calcium carbonate was provided by OMYA supplier (reference : Réf : F38052, d_{10} (µm) : 0.7 µm, d_{50} (µm) : 2.5 µm, d_{90} (µm) : 6.5 µm, d_{98} (µm) : 10 µm).

Complementary studies were carried out with a specific transparent cell equipped either a with piece of filter cloth (filtering surface 5x0.6 mm^2) or with a circular piece of filter cloth (radius 1.5mm thickness 0.6 mm) to observe the deposit of the first layers of calcium carbonate on the filter cloth.

Cake discharge

A prototype device has been developed to remove the filter cake from the filter cloth by a perpendicular traction, a tangential shearing or a combined traction (in an oblique direction), respectively. In the present study, only the shearing mode was studied according the principle scheme presented in figure 1. The system cake + filter cloth is located in a bracket and fixed on the prototype base. A specific plier grabs the filter cloth to a part of its periphery and removes it form the cake in the horizontal direction with a speed of 2 mm/s.

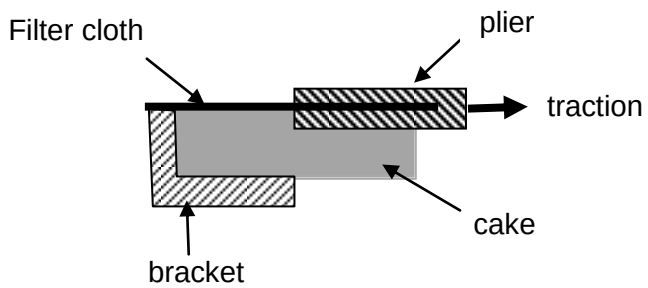

Figure 1 : Principle of filter cloth detachment from cake by shearing

A typical curve obtained during the measurement is illustrated figure 2.

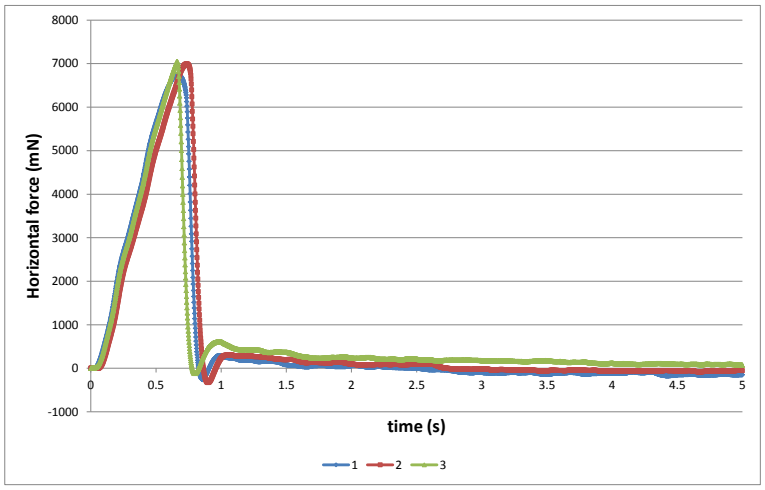

Figure 2 : Calcium carbonate cake detachment from synthetic filter cloth by shearing

The figure 2 enables us to determine the maximal force and the corresponding shear stress by dividing the force by the contact surface between the cake and the filter cloth required to initiate the detachment. The global energy required to achieve a complete detachment is the product of the force and the corresponding displacement. Further measurements such as the mass of residual solids (retained on the filter cloth) and the loss of permeability (of the filter cloth) were also conducted. Tests were carried out at least 3 times and highlighted a satisfying repeatability with a standard deviation below 10%. .

Results and Discussion

Calcium carbonate cake formation

A first series of tests was carried out by increasing concentration of calcium carbonate. A minimal concentration of 33 g/L was necessary to form a filter cake. Preliminary results performed with the transparent horizontal cell at low pressure (0.7 bar) has shown that the first layers deposited on the filter cloth formed a compact layer whereas the other particles were attracted by the first layers but they were labile and did not form any cohesive layers (figure 3).

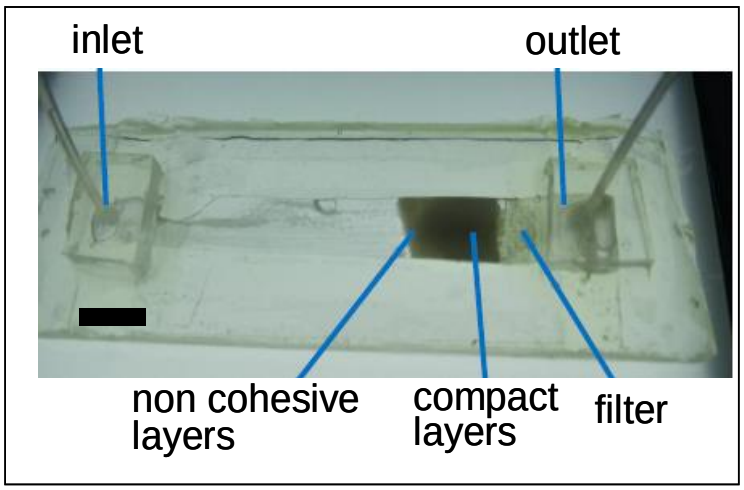

Figure 3: Calcium carbonate cake formation in a horizontal transparent glass cell. The dark scale bar corresponds to 5mm.

The cake formation was also studied in another configuration (figure 4, left) completely saturated with water, with a greater filtering area (7.1 vs 3 mm^2), still fed with a suspension of 33 g/L concentration but with a lower initial pressure (0.1 bar). In this case, the particles settle within the tubing at the beginning of the experiment. As a result a dense suspension pocket is formed very close to the inlet of the cell (figure 4a) and then it flows towards the filter cloth. In the following, this dense pocket clogs the filter. It's worth noting that until the end of the test the concentration of the feeding suspension remains constant, i.e., no other dense suspension pocket is formed anymore. The cake formed on the filter is compacted over time in a linear manner (figure 4, graph on the right) under constant pressure of 0.1 bar. When the height of the cake stops to grow, the applied pressure is increased up to 0.2 bar which leads to slightly steeper compaction rate. Finally, another increase of the pressure, up to 0.4 bar, ends the squeezing process. We get a cake compression of around 400 % from the cake formation up to the end of the test.

These observations are a help for the understanding of the physical mechanisms of the cake formation. For the same cake thickness, the manner the layers of solids are deposited on the filter cloth may contribute to the behavior of the cake during its discharge.

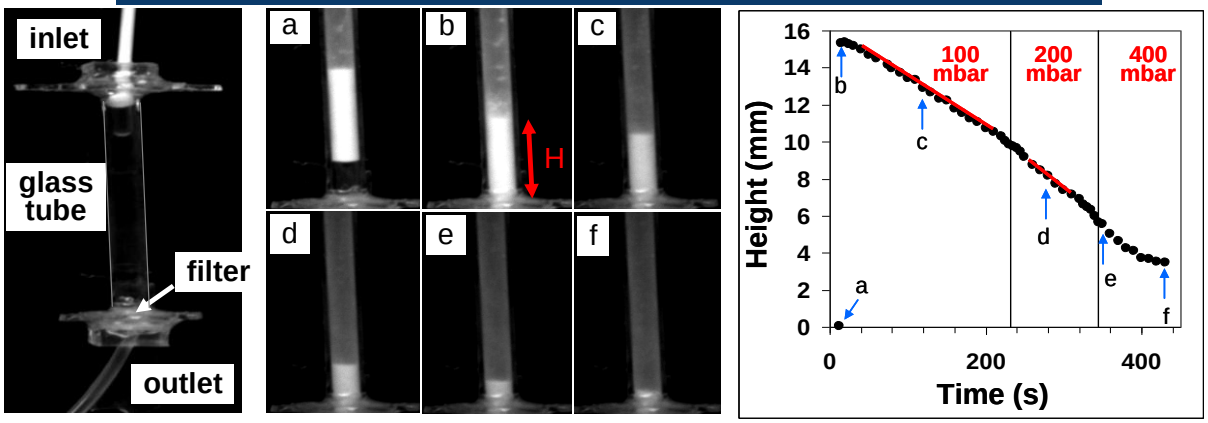

Figure 4: (left) Calcium carbonate cake formation in a vertical transparent glass cell (inner diameter 3mm). (middle) consecutive images (*a* to *f*) of the cake formation. In image *a*, a dense pocket of suspension flows towards the filter and get into contact with it few seconds later (image *b*) and delayed the initial time of cake formation. This event corresponds to the highest point in the graph on the right. Evolution of the height of the cake (delimited by the arrow) with time for three consecutive pressure step (100, 200 and 400 mbar).The two red lines correspond to linear fits of the data.

Results of filtration-compression tests obtained in a vertical filtration-compression cell with the same filter cloth (REF 1, polypropylene, monofilament/staple, bubble point : 6 µm) at different pressures are presented in table 1.

Table 1 : Results of filtration-compression tests of calcium carbonate (50 g at 250 g/L) at different pressures with filter cloth (ref 1).

Pressure (bar)	5	8	10	13
Filtration time (min)	20	13	11	9
Specific resistance (10^{11} m/kg)	5.4	5.7	6	6.4
Cake thickness (mm)	9	8	8	8
Cake dryness (%)	80.6	80.6	80.8	81.3
Ratio of filter cloth permeability before and after test (%)	0.062	0.112	0.221	0.250

The results highlight that the calcium carbonate cake has a medium specific resistance to filtration and a low compressibility. The increase of pressure between 5 and 10 bar allows a decrease of filtration time and fouling of the filter cloth without any significant change of the cake thickness and dryness.

Discharge of calcium carbonate cakes obtained different pressures

The cakes, obtained at different pressures (table 1), were discharged from the filter cloth (ref 1) with the prototype device (figure 1). Results are presented in table 2.

Table 2 : Results of filter cloth (ref 1) detachment from calcium carbonate cakes formed at different pressures

Pressure (bar)	5	8	10	13
Maximal shear stress (Pa)	4025±330	4050±170	4980±440	7050±440
Detachment energy (J/mm^2)	28.6±8.96	35.4±6.14	21.0±1.21	14.4±1.87
Solids loss on filter cloth (%)	1.45±0.12	1.52±0.13	1.00±0.17	1.07±0.18

The increase of pressure between 5-8 and 10-13 bar during filtration-compression involved a higher stress to initiate the cake detachment (that means that the cake discharge by gravity may need assistance by mechanical means) but a lower energy to achieve the complete cake discharge. This behaviour can be explained by the fact that the filter cloth contained less solids (and was consequently less clogged). The rupture of bridges between particle layers and filter cloth were more complete and total at 13 bar than at 5 bar. Consequently, the quantity of cleaning liquid might be lower in this situation.

Discharge of calcium carbonate cakes formed at 10 bar from different filter clothes

Series of experiments were carried out for monofilament filter clothes manufactured with different textile material. The other parameters such as the thread diameter, the weaving type and the permeability were similar. Results are presented in table 3.

Table 3 : Results of carbonate calcium detachment from equivalent filter clothes only differing by the textile material

Textile material	Polyamide 12	Polypropylene standard	Polypropylene special
Maximal shear stress (Pa)	2225±140	4338±720	6026±245
Detachment energy (J/mm^2)	14.5±0.85	40.6±4.48	30.9±4.79
Solids loss on filter cloth (%)	0.883±0.061	0.573±0.087	0.293±0.030

Results show that polyamide material allows an easier cake discharge than polypropylene material even if a higher quantity of solids remains trapped in the filter cloth. The main advantage of special polypropylene comparing to standard propylene was the decrease of the detachment energy and of residual solids (even if a higher shear stress was necessary to initiate cake detachment).

Another series of tests were carried out with two polypropylene monofilament filter clothes woven in the same time with the same thread and with the same bubble point (18 µm). They just differed with regard to their pattern : twill (weft thread passing over or under 2 or more warp yarns) or satin (smooth surface). Results are presented in table 4.

Table 4 : Results of carbonate calcium detachment from equivalent polypropylene filter clothes only differing by the surface treatment

Surface treatment	Satin	Twill
Maximal shear stress (Pa)	4957±342	6346±367
Detachment energy (J/mm^2)	19.6±2.13	18.2±1.1
Solids loss on filter cloth (%)	0.320±0.04	0.56±0.06

Results show that filter cloth with satin surface treatment lead to a lower shear stress to initiate cake detachment comparing to twill filter cloth. This behavior may be due to a lower quantity of solids trapped inside the filtering medium as spacings between points of interlacing are wider.

Conclusions

An experimental method has been developed to quantify the adhesion of filter cakes on filter clothes. Some works are still under progress to improve the reliability and the repeatability of the measurement. The paper presented the influence of two important parameters (pressure and filter cloth properties) which contribute to cake stickiness to the filter cloth and modify its discharge behavior. In order to better understand the interaction between particles and filter cloth, a filtering medium of controlled characteristics will be manufactured by lithography. This work will help to optimize cake discharge time by choosing the most adapted operating conditions for a fast cake discharge in industrial devices. The development of a cake discharge index is foreseen to ease the comparisons between suspensions of different origins and different operating conditions.

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