Batch Filtration Studies of Fine Calcites

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Cake filtration is mainly used as a solid-liquid separation unit operation in the mineral industry. It is used to recover or to remove a maximum possible quantity of liquid from the thickened slurries and to produce a cake at the upstream side of a filter media under an applied pressure difference. The characteristics of the cake structure are the key factors which influence filtration process and its results. This research study is intended to investigate the batch filtration parameters of fine calcite samples from Brandenburg using a pressure filter machine. The filtration parameters were optimised according to the filtration time, the cake thickness, the filtrate density and the humidity loss ratio against change in filtration pressure. The shortest filtration time was determined as t = 206 s under $\gamma = 500$ g l⁻¹ suspension concentration and $\Delta p = 3.5$ bar filtration pressure. The cake thickness was found to be h = 7.92 mm. under $\gamma = 500$ g l⁻¹ suspension concentration and $\Delta p = 0.8$ bar filtration pressure. The highest humidity loss mass fraction measured as w = 21.38 % under $\gamma = 250$ g l⁻¹ suspension concentration and $\Delta p = 0.8$ bar filtration pressure. The lowest filtrate density was measured as $\rho = 0.995666$ g ml⁻¹ under 500 g l⁻¹ suspension concentration and $\Delta p = 3.5$ bar as a filtration pressure.

Key words:

Batch filtration, fine calcites, solid-liquid separation, dewatering

Introduction

Filtration is an important method of solid-liquid separation in chemical, metallurgical, food and environmental industries. Conventional cake filtration is difficult when the size of solid particles becomes finer. The finest clog of the pores of the filter medium, thereby negatively affects the filtration rate. Filter media clogging may occur as a reversible or irreversible phenomenon depending on particulate and the media characteristics.^{1,2,3,4}

Filtration in the mineral processing industry can be divided into three main categories: batch filtration, continuous filtration, and clarification. The applied theory of these systems is different from each other. The methods of operation are generally considered in batch filtration with constant pressure and constant volumetric flow rate filtration. In actuality, the batch filter cycle may be either a combination of the two, or modifications of both, depending on filter feed pump selection. However, it is most convenient to obtain equations by employing experiments using either or both of these methods.⁵

The process of filtration is often related to the flow of a fluid through a bundle of capillary tubes in a filter cake. In this case, one can use the Poisseulle's equation derived in 1846. The basic expression most commonly employed as a starting point is the Poisseulle's equation;

$$\frac{\mathrm{d}V}{\mathrm{d}t} = \frac{\Delta PA}{\eta \left(a\gamma \frac{V}{A} + R\right)} \tag{1}$$

where the V is volume of filtrate; A is a area of filtration; t is time; Δp is pressure drop across the filter (including filter cloth and drainage network), η is a liquid viscosity; a is a specific cake resistance; γ is mass of dry cake solids per unit volume of filtrate, and R is resistance of filter cloth and drainage network, A/V. Rearranging the equation,

$$\eta a \gamma V \frac{\mathrm{d}V}{A^2} + R \frac{\eta \,\mathrm{d}V}{A} = \Delta p \,\mathrm{d}t \tag{2}$$

If constant pressure filtration is assumed and temperature and feed conditions are kept constant, $\eta\gamma a$, and Δp are also constant. The term a is supposedly a function only of Δp and therefore considered constant also. Accordingly, by integration,

$$\frac{\eta a \gamma V^2}{2A^2} + \frac{\eta R V}{A} = \Delta p t \tag{3}$$

Dividing the equation by V/A and Δp and rearranging.^{5,6,7}

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$$\frac{t}{V/A} = \frac{\eta a \gamma V}{2\Delta p A} + \frac{\eta R}{\Delta p} \tag{4}$$

Dewatering of fine minerals can be described as a process associated with the flow of capillary water through the porous media created by a bed of particles on the filter media. Darcy also derived the rate equation of a dewatering process as follows;

$$\frac{\mathrm{d}V}{\mathrm{d}p} = k\frac{A}{\eta}\frac{\Delta p}{h} \tag{5}$$

where the *V* is the volume of fluid, *t* is the filtration time, Δp is the pressure drop across the filter cake, *h* is the cake thickness, *A* is the cross-sectional area of the cake, η is the viscosity of the liquid, and *k* is the rate constant known as a cake permeability. According to equation (5), the rate of dewatering is proportional to the pressure drop across the cross sectional area, and it is inversely proportional to the viscosity of the process water.^{6,8,9}

In filtration, the important forces are the force acting on the particle from the filter media and the fluid friction. As the particles form a packed bed the resistance to flow in the bed becomes more important than the resistance in the filter media. In practical applications the flow of the fluid is laminar, allowing the use of the Carman-Kozeny correlation;

$$\frac{\Delta p}{h} = 36k \frac{(1-\varepsilon)^2}{\varepsilon^3} \frac{\eta_e v}{d_p^2}$$
(6)

where the Δp is the pressure drop across the filter cake, *h* is the cake thickness, ε is the void fraction, η_e is the effective viscosity, *v* is the velocity of the solid particles and d_p is the particle diameter. By defining α as the specific cake resistance, *R/m*:

$$\alpha = \frac{5(1-\varepsilon)A_0}{\rho_s e^3 V^2} \tag{7}$$

(k = 5 only valid for balls, as constant)Equation (6) can be expressed as;

$$\frac{\mathrm{d}t}{\mathrm{d}V} = \frac{\eta\alpha\gamma V}{A^2(-\Delta p)} \tag{8}$$

$$\Delta p = \rho g_{\rm c} h \tag{9}$$

Equation (8) can be expressed as;

$$\frac{\mathrm{d}t}{\mathrm{d}V} = \frac{\eta \alpha \gamma V}{A^2 (-\rho g_{\mathrm{c}} h)} \tag{10}$$

Where the g_c is the gravitation constant and this equation is a straight line relation between dt/dV and V if $-\Delta p$ is held constant. Thus, from actual constant pressure filtration data dt/dV may be plotted as a function of *V*. From the slope of the line α can be evaluated.¹⁰

Cake dewatering is a post-filtration process in which the filtrate trapped in the pores of filter cakes is removed by some mechanical or hydrodynamic means to save energy in thermal drying or to improve handling qualities of filter cakes. Mechanical means are used to compress the cake and bring the particles closer in order to squeeze out the excess liquid. Displacement of the retained liquid by sucking or blowing air through the cake, the subject of this paper, is probably the most common method of cake dewatering in the mineral and coal industry.¹¹

A minimum driving pressure difference across the cake, called the entry pressure, is required for the displacement of liquid by air at the filter cake surface. Once this pressure is overcome, the cake dewaters at a gradually decreasing rate, and the cake moisture, or more fundamentally the cake saturation, defined as the fraction of pore volume filled with liquid, approaches to an irreducible level (residual saturation) for a given set of operating conditions.¹²

The residual saturation results from a state of equilibrium between the driving pressure differential and the moisture-retaining capillary forces. There are two distinct regimes through which filter cakes are dewatered to their residual saturation levels, these are:

(i) no-air-flow regime in which the cake is partially saturated, but no air breakthrough occurs through the filter cake and filter medium;

(ii) air-flow regime in which air breaks through the filter cake and the filter medium, which corresponds to residual saturation levels less than about 0.60–0.64 under normal vacuum-filtration conditions. The key factors that will determine which dewatering regime to prevail are the pore sizes of, both, the cake and the filter medium, and the applied pressure differential.^{13, 14}

In the beginning of the filtration, mineral particles are passed through the filtrate since the radius of the solid particles is smaller than the pore diameter of the filter cloth, until the particle bridge is occurring. This case is illustrated in Figure 1.¹⁵

In this study, the batch filtration data of fine calcite samples from Brandenburg were investigated in detail. The important filtration parameters were selected as applied pressure, time, humidity loss, filtrate density, and the thickness of the filtered cake, in order to explain the roles of cake compression and filter-cake media interface on the cake filterability, as the characteristics of the cake structure are the key factors which influence filtration process and its results. The aim of the batch fil-



Fig. 1 – Occuring of the particle bridge on the filter cloth

tration optimisation was to find out the shortest filtration time, the highest cake thickness, the lowest filtrate density and the highest humidity loss ratio against change in filtration pressure.

Materials and methods

In the experimental work, representative calcite samples in pulp form from Brandenburg were experimented for determination of their filtration behaviour by using a batch pressure filter machine. Experimental quantities were filtration pressure, filter cake thickness, suspension concentration and residence time. Figure 1 shows the particle diameter distribution of calcite samples which was used in the filtration tests. According to the Figure 2, the mean particle diameter was determined d_{50} : 72 μ m and d_{80} : 122 μ m.



Fig. 2 – Particle diameter distribution of calcite samples

Pressure filtration machine with a litre capacity and a suitable filter media for laboratory was used in the filtration tests in order to determine the time, the mass, and the density of the calcite samples dur-



Fig. 3 – Pressure filtration test equipment used in the experiments

ing filtration. A digital balance was also used for determination of filtration quantities as shown in a schematic diagram of the filtration system modified from¹⁶ in Figure 3.

Filtration tests

Several filtration tests were carried out considering the following quantities; suspension concentration, desired cake thickness, residence time, filter type, and pressure. These quantities were quantified with suitable analytical techniques from a previous study. All experiments were performed with a previously defined $\tau = 50$ s residence time and with a suitable nylon filter media.¹³ Figure 4 illustrates the variables modified from¹⁷ which affect the filtration process.

In the batch filtration tests, the initial suspension concentrations (500-250-170 g 1^{-1}) against different filtration pressures (0.8–2.2–3.5 bar) were experimented in order to reveal the optimal values of filtration time, filter cake height, humidity loss, and filtrate density. The optimised results are given in Figure 5,6,7 and 8 as separate graphs.

Batch pressure filtration experiments were carried out under following conditions;

- 500 g l^{-1}, 250 g l^{-1}, 170 g l^{-1} of suspension concentrations,
- -0.8 2.2 3.5 bar of positive pressures,
- Filter media : Nylon,
- 8-5-3 mm of desired filter cake thicknesses,
- 50 s of residence time.



 $\begin{array}{ll} V_{g,e} = \text{ suspension volume } \rho_o = \text{ suspension density } \\ V_{g,a} = \text{ filtrate volume (passing from a pore)} \\ V_{La} = \text{ filtrate volume } \rho_u = \text{ filtrate density } \\ R_1 = \text{ resistance of the filter cake } h_K = \text{ cake height } \\ R_2 = \text{ resistance of the filter cloth } h_{KE} = \text{ substitute cake height } \end{array}$



In Figure 5, it is clear that filtration time sharply decreases when the pressure is increased for all initial suspension concentrations. However, residence time decreases at lower rate, when initial suspension concentrations get higher.



Fig. 5 – Variation of filtration time against different initial suspension concentrations and filtration pressures

From Figure 6, it is seen that filter cake thickness can be reduced from 8 to 7 mm when higher pressures and lower initial suspension concentration are applied during filtration. However, when higher initial suspension concentrations are used, increase in filtration pressure does not really affect the filter cake thickness.

Figure 7 shows that humidity loss is not necessarily affected by change in filtration pressure and initial suspension concentration.



Fig. 6 – Variaton of filter cake thickness against different initial suspension concentrations and filtration pressures



Fig. 7 – Variation of humidity loss against different initial suspension concentrations and filtration pressures



Fig. 8 – Variation of filtrate density against different initial suspension concentrations and filtration pressure

As it is clearly seen from Figure 8, increasing the filtration pressure and initial suspension concentration is not necessarily effective for the change in the filtrate density. According to the related Figures 5, 6, 7, and 8, the optimal filtration results can be summarized as below;

The shortest filtration time was determined as 206 s under 500 g l⁻¹ suspension concentration and 3.5 bar filtration pressure as clearly seen from Figure 5. From Figure 6, the cake thickness was found to be 7.92 mm. under 500 g l⁻¹ suspension concentration and 0.8 bar filtration pressure. The highest humidity loss fraction measured was as 21.38 % under 250 g l⁻¹ suspension concentration and 0.8 bar filtrate density was measured as 0.995666 g ml⁻¹ under 500 g l⁻¹ suspension concentration and 3.5 bar pressure filtration pressure.

Discussion and conclusions

Residence time was increased due to the high amount of the filtrated liquid in the lower suspension concentrations, thus decreasing of the suspension concentrations was affected by increasing of the filtration time.

Because of the high suspension concentration of the pulp, the cake thickness was increased due to inclusion of the high amount of the solid particles.

Humidity loss curves of the 170 g l^{-1} and 250 g l^{-1} suspension concentrations were intersected in 1 and 3 bar pressures, for this reason we considered, that the humidity loss was related with the filtration pressure rather than the concentration of the pulp.

These results explains, that increasing the pressure will proportionally increase the power consumption in laboratory conditions, while decreasing the pressure will affect the filtration performance and therefore filtration process will take longer. As a result, 500 g l⁻¹ concentration and 2.2 bar pressure are chosen as the optimal values for suitable power consumption, filtration speed and efficiency for this wash. In industrial applications, the lowest power consumption and filtration time should be taken into account, when optimisation of the batch filtration results was performed. These findings can only give a brief evaluation of batch filtration studies depending on the material properties, however, when the professional chemical engineering application are considered, not only material properties but also sufficient continuous filtration methods and working parameters should be investigated.

List of symbols

- A area of filtration, cm^2
- *a* specific cake resistance, R/m, g^{-1} cm⁻¹
- dp particle diameter, μm

- h cake height, cm
- g_c gravity acceleration constant, ms⁻²
- k cake permeability, cm²
- m mass, g
- Δp pressure difference, bar
- R cake resistance, A/V, cm¹
- t filtration time, s
- V suspension volume, 1
- v velocity of particle solid, cm sn⁻¹
- w mass fraction of humidity loss, %
- γ suspension concentration, g l⁻¹
- ε void fraction, %
- η liquid viscosity, kg m⁻¹ s⁻¹
- ρ liquid density, g l⁻¹
- τ residance time, s

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