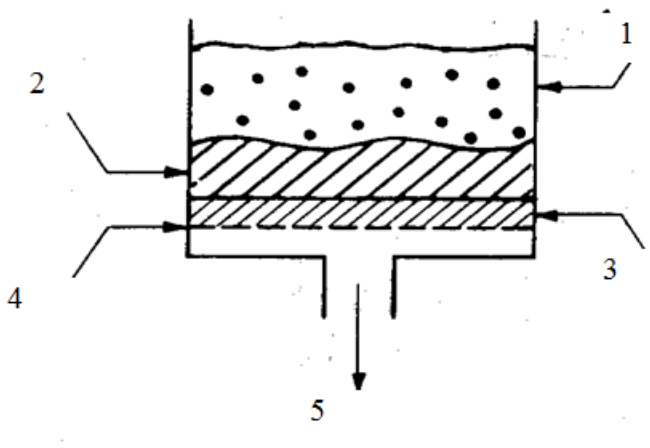
Filtering and centrifugation

The aim of the measurement is to gain practical knowledge about a hydrodynamic separation process used to purify suspensions.

1. Theoretical background

Filtration is a hydrodynamic separation process, the driving force of which the separationis pressure difference. Selectivity is initially given by the filtering medium, but during the filtration filtering cake plays the major role. It can be used in the separation of liquid-solid systems (suspensions), gas-solid systems (air contaminated with dust) or gas-liquid systems.

During the filtration of liquid-solid systems, suspensions are forced through a porous layer of particles (the filtering medium) that holds back floating particles and lets liquids pass through. Its schematic depiction of a simple filtering is shown in Fig. 1.



1. Figure – A schematic depiction of the filtration process. 1 Suspension, 2 Slurry layer (cake), 3 Filter medium, 4 Grid, 5 Filtrate

1.1 Resistance of the deposited layer

In 1830, *Darcy* studied the filtration velocity of water through a layer of sand. The measurements proved that the filtration rate, which is the volume of the filtrate obtained over a unit of time in a unit of filtration surface, is proportional to the pressure difference, but it is inversely proportional to the viscosity and of the liquid and to the thickness of the deposited layer.

$$v = \frac{1}{A} \cdot \left(\frac{dV}{dt}\right) = B \cdot \frac{\Delta p_l}{\eta \cdot l} \qquad (1)$$

where v $\left[\frac{m}{s}\right]$ denotes the filtration rate, V $[m^3]$ is the volume of the filtrate, A $[m^2]$ is the surface area of the filter, η $[Pa \cdot s]$ is the dynamic viscosity of the liquid, l $[m]$ is the thickness of the filter medium, t $[s]$ is the filtration time, Δp_l $\left[\frac{N}{m^2}\right]$ is the pressure difference between the two sides of the filter and B $[m^2]$ is the coefficient of permeability of the filtering medium. It must

be noted, that the equation is only valid for non-compressible filtration cakes and Newtonian fluids.

The formula (1) is in good accordance with the theory of a laminar flow through a layer of particles, more precisely with the *Blake-Kozeny* equation. Deriving the mean velocity:

$$v = \frac{1}{A} \cdot \left(\frac{dV}{dt}\right) = \left[\frac{\varepsilon^3}{K \cdot (1-\varepsilon)^2 \cdot \omega_p^2}\right] \cdot \frac{\Delta p_l}{\eta \cdot l} \qquad (2)$$

from which, by the reduction of the expression in brackets and substitution of B, the *Darcy*-equation can be obtained. It the formula (2), K is a constant value, ω_p $\left[\frac{m^2}{m^3}\right]$ is the specific surface area of the particles and ε $\left[\frac{m^3}{m^3}\right]$ is the voidage.

The pressure drop can be expressed from equation (1).

$$\Delta p_l = \frac{\eta}{A} \cdot \frac{l}{B} \cdot \left(\frac{dv}{dt}\right) \qquad (3)$$

Instead of the $\frac{l}{B}$ resistance of the slurry layer, which has the unit $[m^{-1}]$, it is common to use

$$\frac{l}{B} = \frac{(\alpha \cdot c \cdot V)}{A}$$

where α $\left[\frac{m}{kg}\right]$ is the specific resistance of the slurry and c $\left[\frac{kg}{m^3}\right]$ is the mass of the particles deposited from a unitary volume of filtrate.

$$\Delta p_l = \frac{\eta}{A} \cdot \left(\alpha \cdot c \cdot \frac{V}{A}\right)\left(\frac{dV}{dt}\right) \qquad (4)$$

It is more practical to use the expression $\frac{\alpha \cdot c \cdot V}{A}$ instead of the resistance of the slurry layer $\frac{l}{B}$ because the dependence of the resistance of the V filtrate volume is included: the resistance increases with the increasing volume. This also means, that the thickness of the slurry layer l can be expressed from the mass balance constructed for the layer:

$$l \cdot A \cdot (1-\varepsilon) \cdot \rho_p = c \cdot (V + \varepsilon \cdot l \cdot A)$$

where ρ_p $\left[\frac{kg}{m^3}\right]$ is the density of the particles. The last addend is the volume of the filtrate trapped in the slurry. This is negligible, thus equation (5) is a good approximation.

$$l = \frac{c \cdot V}{A \cdot (1-\varepsilon) \cdot \rho_p} \qquad (5)$$

Using the equations (2) and (5) we can get

$$\frac{dV}{dt} = \frac{\Delta p_l \cdot A}{\eta} \cdot \left(\frac{\varepsilon^3 \cdot \rho_p}{K \cdot (1-\varepsilon) \cdot \omega_p^2}\right) \cdot \left(\frac{A}{c \cdot V}\right)$$

and, substituting the α specific resistance:

$$\alpha = \frac{K \cdot (1-\varepsilon) \cdot \omega_p^2}{\varepsilon^2 \cdot \omega_p^2} \qquad (6)$$

Equation (2) transforms into equation (4):

$$\frac{dV}{dt} = \frac{\Delta p_l \cdot A}{\eta \cdot \left(\alpha \cdot c \cdot \frac{V}{A}\right)}$$

1.2 The resistance of the filter medium

During filtration, to force the filtrate through the filter, it is necessary to take into account other resistances besides the resistance of the deposited layer which effects also increase the pressure drop. These are:

- the resistance of the filter medium (the filter cloth) and
- the resistance of the piping and the accessories of the filtering apparatus.

The latter two resistances will be handled together and marked with R_m $\left[\frac{1}{m}\right]$.

We can construct the equation for the pressure drop of the filter medium p_m in a similar way to equation (4):

$$\Delta p_m = \frac{\eta}{A} \cdot R_m \left(\frac{dV}{dt}\right) \tag{7}$$

The total pressure drop on the equipment is the sum of Δp_l and Δp_m:

$$\Delta p = \Delta p_l + \Delta p_m = \frac{\eta}{A} \cdot \left(\frac{dV}{dt}\right) \cdot \left(\alpha \cdot c \cdot \frac{V}{A} + R\_m\right) \tag{8}$$

Rearranging equation (8) leads to the so called *Carman*-equation of filtration:

$$\frac{dV}{dt} = \frac{\Delta p \cdot A}{\eta \cdot \left(\alpha \cdot c \cdot \frac{V}{A} + R_m\right)} \tag{9}$$

Performing filtration at a constant pressure, equation (9) can be integrated:

$$\int_0^t dt = \frac{\eta}{A \cdot \Delta p} \cdot \left(\frac{\alpha \cdot c}{A} \cdot \int_0^V V\, dV + R_m \cdot \int_0^V dV\right) \tag{10}$$

thus

$$t = \frac{\eta}{\Delta p} \cdot \left[\frac{\alpha \cdot c}{2} \cdot \left(\frac{V}{A}\right)^2 + R_m \cdot \frac{V}{A}\right] \tag{11}$$

And, from this, the volume of the filtrate:

$$V = \frac{A}{\alpha \cdot c} \cdot \left(\sqrt{R_m^2 + \frac{2 \cdot \alpha \cdot c \cdot t \cdot \Delta p}{\eta}}\right) - \frac{A \cdot R_m}{\alpha \cdot c} \tag{12}$$

1.3 Determination of the filtration constants

Equation (9) can also be constructed as

$$\frac{dV}{dt} = a \cdot V + b \tag{13}$$

where

$$a = \frac{\alpha \cdot c \cdot \eta}{A^2 \cdot \Delta p}$$

is the slope of the linear function while

$$b = \frac{R_m \cdot \eta}{A \cdot \Delta p}$$

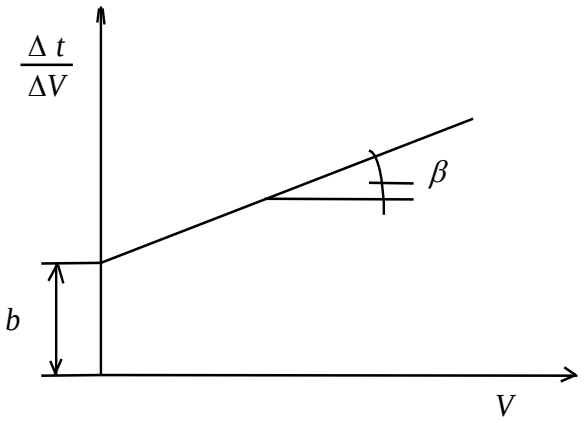
is the intersection of the line with the vertical axis.

If we assume that

$$\frac{dV}{dt} \cong \frac{\Delta V}{\Delta t} \tag{14}$$

a diagram can be prepared from the measurement, where $\frac{\Delta t}{\Delta V}$ is plotted against the V mean filtrate volume. Using the linear fitted to the measured points, the filtration constants ($\alpha \cdot c$ and R_m) can be determined from its a slope and b axial intercept.

If you measure, that between times t_1 and t_2 the volume of the filtrate increased from V_1 to V_2, $\Delta t=t_2-t_1$, $\Delta V=V_2-V_1$ and the calculated ratio has to be plotted at volume $V=(V_1+V_2)/2$.



2. Figure – Determination of the filtration constants.

1.4 Determination of the optimal filtration time

Let us mark the longest possible filtration time with t_{max}. It is determined by two factors:

1) the quantity of the suspension to be filtered (V_0 total filtrate volume)
2) the fact that the volume of the slurry in the filter is limited ($V_{slurry_{max}}$):

$$V_{slurry_{max}} = l_{max} \cdot A = k \cdot V \qquad (15)$$

where l_{max} denotes the maximal thickness of the slurry layer, V is the volume of the liquid already filtered and k is a constant. Quite often, the total filtrate volume we have to obtain is much larger than the right side of the equation (16).

$$V_{total} \gg \frac{V_{slurry_{max}}}{k} \qquad (16)$$

In such a case, the suspension needs to be filtered in multiple fractions. However, it is not beneficial to determine the quantity of filtrate to be processed using equation (15) and the maximal volume of the slurry, as with the thickening of the cake its resistance also increases, and the filtration rate defined in equation (1) decreases. It can be easily understood that a lower rate decreases the efficiency of filtration. Because of this, the actual filtration time is usually chosen to be lower than t_{max}.

The time needed to perform a complete filtration period, the total time (t_{total}) can be divided into two parts.

- to a so called downtime or dead time (t_d) that includes washing, removing the filter cake, washing of the filter cloth and, in case of frame filter presses, the time needed for the assembly and disassembly of the equipment
- the filtration time (the time the equipment is operating) (t)

If V_{total} is significantly larger than $\frac{V_{slurry_{max}}}{k}$, then, during the determination of the optimal filtration time, we want to determine the maximum of the filtrate obtained during a unitary amount of time, thus the maximum of the following equation:

$$\frac{V}{t_{total}} = \frac{V}{t_d + t} \tag{18}$$

The relation of the filtrate volume and the filtration time is described by the equation (12). Let us mark the functional relation between V and t_{total} with

$$V = f(t_{total}) \tag{19}$$

Equation (18) has an extremum where:

$$\frac{d\left(\frac{V}{t_{total}}\right)}{dt_{total}} = \frac{d\left(\frac{f(t_{total})}{t_{total}}\right)}{dt_{total}} = 0 \tag{20}$$

the derivative of the fraction expression:

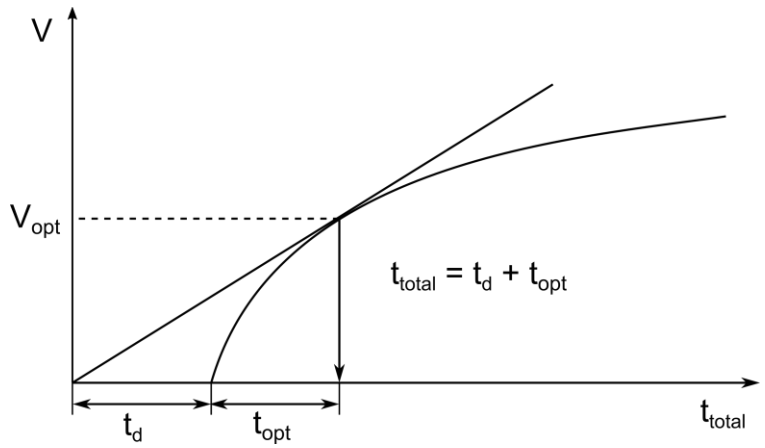
$$\frac{d\left(\frac{f(t_{total})}{t_{total}}\right)}{dt_{total}} = \frac{t_{total} \cdot f'(t_{total}) - f(t_{total})}{t_{total}^2} \tag{21}$$

Thus,

$$f'(t_{total}) = \frac{f(t_{total})}{t_{total}} \tag{15}$$

That means that at the optimum, the derivative equals the fraction expression

$$\frac{V}{t_d + t_{opt}}$$



3. Figure – Graphical determination of the optimal filtration time.

The equation above is most conveniently solved graphically. Let us plot the volume of the filtrate against the total time (t_{total}) (Figure 3.). Then, we draw the contingence line to this curve from the origin, which means the maximal slope corresponding to the optimal filtration time and marks the maximum filtration performance $\left(\frac{V}{t_{total}}\right)$.

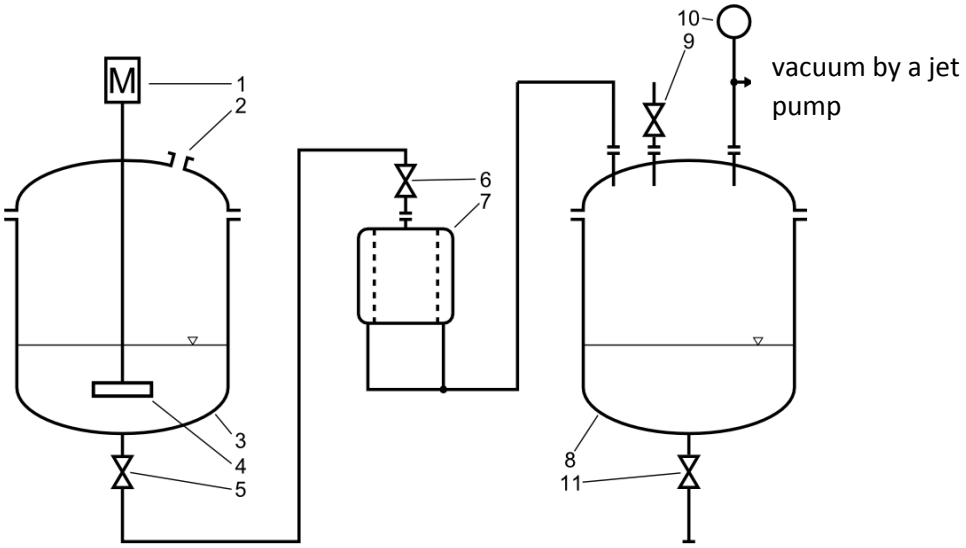
The method described above can be used in finding the optimum of any kind of batch process, if the price of the product is determined by the manufacturing time.

2. The description of the filtration measurement

The aim of the measurement: the purification of a suspension of grained perlit using a frame filter press. Determination of the optimal filtration time and the optimal filtrate volume using

the graphical method. Comparing the values obtained by the graphical method to those obtained by calculation.

The flowsheet of the laboratory filter press used in the practice is shown in Figure 4.



4. Figure – 1 Stirring motor, 2 Admitting port, 3 Tank, 4 Stirrer, 5 Tap, 6 Tap, 7 Filter press, 8 Filtrate tank, 9 Tap for pressure regulation, 10 Pressure gauge, 11 Tap

Before starting the measurement, make sure that the taps number 5 and 11 are closed and the taps number 6 and 9 are opened. You will find the filter press assembled and ready for the measurement. Anyway, please check it before use. After connecting the lines, the filter press needs to be turned by 90°, to the position shown in Figure 4.

Suspend 100-150 g of dry Perfil 250 (grained perlite) in 10 dm^3 of water. Mix the suspension before filling it into the feeding tank. Start stirring. Open the tap of the jet pump and close the tap number 9. Wait for the system to reach the vacuum value given by the supervisor (0.25-0.6 bar of pressure difference). After reaching the desired vacuum value, filtration can be started by opening the tap number 5. A stopwatch has to be started at the same time. Measure the time needed to collect 0.5; 1; 1.5 l….etc. of filtrate. This can be accomplished using the scale at the side of the tank number 8. The desired vacuum can be maintained by setting up the tap number 9. Perform filtration until the tank number 3 runs out of suspension. Then, air should be driven through the system for another 3 minutes, so that the perlite collected is drier, and easier to handle. At the end of the measurement separate the tank number 3 from the filter press over the valve number 6. Fill 2-3 l of water into the tank, stir shortly, then empty the tank. This is necessary to clean the tank and the piping. Slowly open the tap number 9 and close the tap of the water injection pump. After reaching atmospheric pressure, disassemble the filter press and collect the perlite from the filter clothes. The wet filter cake should be placed on the metal plate on the table and placed in the drying cabinet. Wash the parts of the filter press. Measure the diameter of the filter cloth and then assemble the filter press. Take special care about the correct order of its elements. Drain the filtrate into the storage vessel. Measure the time between the end of the filtration (collecting 9 l of filtrate) and the next operation-ready state as well, this will be the downtime. Record the measured data according to the following measurement datasheet.

Measurement datasheet

Area of the filter cloth (A):

Pressure difference (Δp):

Downtime (t_d):

Approximately 20 lines are needed in the table:

V [unit]	t [unit]

3. Evaluation of the measurement results

1. Calculate the two filtration constants $(\alpha \cdot c)$ and R_m. During the calculation, take special care of using appropriate units. During the calculation of filtration constants, summarize the corresponding $\frac{\Delta V}{\Delta t} - V_{mean}$ pairs and plot $\frac{\Delta V}{\Delta t}$ against V_{mean}. Represent the equation of the linear fitted to the measurement points.
2. Determine the optimal filtration time using a graphical method and the also the optimal filtrate volume corresponding to it. (If the contingence line cannot be drawn due to the too long downtime, perform the graphical method and the calculations with an estimated downtime of 10 min.)
3. Determine the optimal filtration time and the optimal filtrate volume by calculation using the filtration constants.

$$V_{opt} = A \cdot \sqrt{\frac{2 \cdot \Delta p \cdot t}{\eta \cdot \alpha \cdot c}}$$

$$t_{opt} = t_d + R_m \cdot \frac{\eta}{\Delta p} \cdot \sqrt{\frac{2 \cdot \Delta p \cdot t}{\eta \cdot \alpha \cdot c}}$$

If the batch time for the optimal filtrate volume is calculated, the downtime needs to e added to the optimal filtration time. Compare the values obtained by the graphical and the calculation methods.

Do not forget captions and the units in the diagrams.

The contents of the report to be submitted

Only present the measurement process and the drawing of the equipment if some difference was discovered compared to the process described before. Otherwise, just a sentence stating that the measurement was done according to the notes, is enough.

1. Measurement datasheet with the signature of the supervisor
2. Detailed calculations with the results highlighted
3. A short discussion of the measurement results

The measurement notes were written using earlier work of the working community of the department by Ildikó Kmecz, translated by Márton Kőrösi, ckecked by Edit Székely.