6 Filtration

Alexandra Novotná, Oldřich Holeček
English translation by Michal Kordač

I Basic definitions

Filtration is a method of separation of solid – fluid mixtures. The mixture if pressed through a layer of porous material (filtration membrane), which holds the solid particles and the fluid is allowed to pass through as filtrate. The solid particles are hold by the membrane in a form of filtration cake. The driving force of filtration is pressure difference above the filtration cake and below the filtration membrane. The pressure difference is produced by either external source of pressure (a pump) or as a result of gravitation or centrifugal forces. Filtration is used if the particles have diameter higher than 100 μm.

Amounts of all streams can be determined by material balance. In further text, m'_S will denote mass of suspension before the filtration in an accumulation tank, m_F mass of filtrate, m_K mass of produced filtration cake, m''_S mass of suspension left in the accumulation tank after the filtration, m_Z mass lost during the filtration, x_S weight fraction of solid phase in the suspension, x_K weight fraction of solids in cake and m_{sZ} loses of the solid phase.

The total mass balance is given as:

$$m'_S = m_K + m_F + m_Z + m''_S \tag{1}$$

and the balance of solid phase is:

$$m'_S x_S = m_K x_K + m_{sZ} + m''_S x_S \tag{2}$$

The time of the filtration depends on the rate of filtration, which is defined as volumetric flow rate of filtrate per unit filtration area:

$$v_F = \frac{1}{S_F}\dot{V}_F = \frac{1}{S_F}\frac{dV_F}{d\tau_F} = \frac{dq_F}{d\tau_F} \tag{3}$$

where q_F is the ratio of filtrate volume to the filtration area. The rate of filtration can be calculated from the driving force (difference of pressure on the filtre Δp_F) dividing by filtration resistance, which is a sum of resistances of the cake and the filtration membrane

$$\frac{dq_F}{d\tau_F} = \frac{\Delta p_F}{R_K + R_M} = \frac{\Delta p_F}{\eta(\alpha_K h_K + \alpha_M h_M)} \tag{4}$$

where α is specific resistance and h is height of the cake (index K) or membrane (index M). As the cake height linearly increases with the volume of the filtrate, the equation (4) is usually transformed into a more useful form:

$$\frac{dq_F}{d\tau_F} = \frac{K_F}{q_F + q_M} \tag{5}$$

where K_F and q_M are filtration constants. q_M gives relative resistance of the filtration membrane and the cake, while K_F is a ratio of the filtration pressure to the cake specific resistance and filtrate viscosity:

$$K_F = \frac{\Delta p_F}{\eta_F \alpha_K \frac{\varphi_K - \varphi_S}{\varphi_S}} \tag{6}$$

where the ratio of the volumetric fractions f is the proportionality constant between volume of cake and volume of filtrate.

At constant Δp_F, the filtration equation (5) is integrated starting at the beginning of filtration at t_{F0}, where q_F has a value q_{F0}, to the end of filtration at t_F:

$$\int_{t_{F0}}^{t_F} K_F \mathrm{d}\tau_F = \int_{q_{F0}}^{q_F} (q_F + q_M)\mathrm{d}q_F \tag{7}$$

which leads to:

$$K_F(t_F - t_{F0}) = \frac{q_F^2 - q_{F0}^2}{2} + (q_F - q_{F0})q_M \tag{8}$$

The two constants in the equation (K_F and q_M) can be obtained by a quadratic regression using a simplified form of the equation (8):

$$t_F = \frac{1}{2K_F}q_F^2 + \frac{q_M}{K_F}q_F + \left(t_{F0} - \frac{q_{F0}^2}{2K_F} - \frac{q_M q_{F0}}{K_F}\right) = a_2 q_F^2 + a_1 q_F + a_0 \tag{9}$$

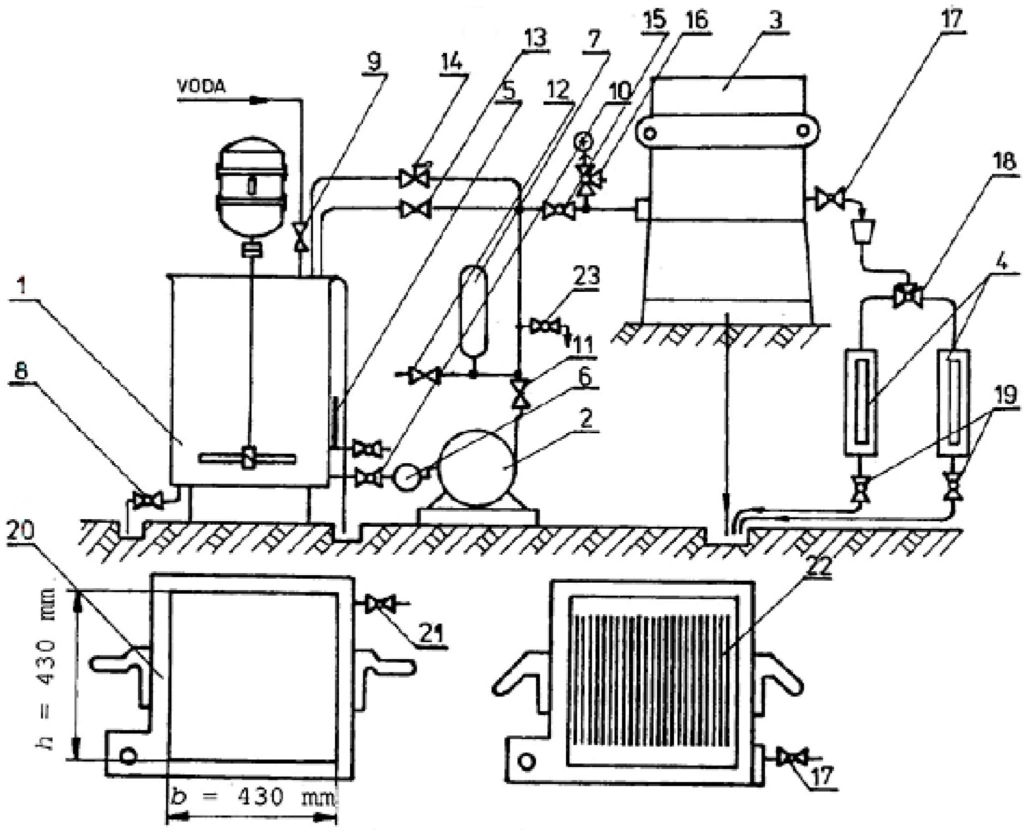
When determining the filtration constants experimentally, it is necessary to prepare the suspension with properties equal to the real system and, also, the suspension for filtration has to be mixed well to prevent sedimentation in some parts of the apparatus. At the beginning of an experiment, the filter is clean and the only resistance to the filtration is due to the filtration membrane. Thus the initial value filtration pressure does not conform with the designed value, which is reached after some of the cake is formed. In this case t_{F0} and q_{F0} are values at the beginning of the constant-filtration pressure period.

II Aims of the work

1. A mass balance of an experimental filtration, determination of filtration cake moist fraction and calculation of possible losses of the suspension and the solid fraction.
2. Determination of filtration constants K_F and q_M from experimental data.
3. Plot a graph of time vs. q_F containing both, experimental values and regression equation. Mind q_F is independent variable while t_F is dependent variable.

III Description of the apparatus

A schema of the apparatus is given on Figure 1.



1 accumulation tank
2 high pressure pump
3 filter press
4 measuring tanks for filtrate
5 liquid level gauge
6 suction filter
7 receiver
8 exhaust valve
9 filling valve
10 suction valve
11 pump exhaust valve
12 valve for tightness test
13 valve for regulating filtration pressure
14 flow rate limiting valve
15 valve opening pipe to filter
16 manometer valve
17 outlet valves of filter
18 filtrate valves
19 measuring tank outlet valves
20 frame of the filter
21 bleeding valve
22 filter plate
23 sampling valve

Filtration test station consists of suspension accumulation tank **1**, multi-stage high pressure pump **2**, filter press **3** and tank for measuring filtrate volume **4**. The accumulation tank is equipped with a turbine impeller and a liquid level gauge, where it is possible to read the suspension volume. The suspension is fed into a pump and recycled back into the tank for better mixing. From this pipeline a side branch leads into the filter press with one frame. At the outlet of the filter the filtrate is collected and led into a pair of tanks with level gauge for measuring volume of filtrate. The volume of the single tank **4** has volume of 40 litres. Which one is used is given by setting the valve **18**.

The frame of the filter **20** has a bleeding valve for removing air **21** and its internal dimensions are 430x430x30mm. The filtration pressure is measured by a manometer, which is on the pipe into the filter. The pressure behind the filtration membrane is equal to the atmospheric pressure.

IV description of work procedure

In this guide, a procedure is given for filtration at constant filtration pressure.

IV.1 Preparation of the suspension.

In the students laboratory, suspension from previous experiments is used. First it is necessary to adjust its volume and then determine the weight fraction of solid phase in the suspension x_S^0. If the weight fraction does not correspond to the task, it is necessary to adjust the concentration.

Volume of the suspension is given by the level gauge **5**. In some cases it is necessary to flush it with water before reading the level. While reading the level, the impeller has to be stopped, as the level in the tank must be flat at this point. If the volume of the suspension is below 400 litres, add water by opening the valve **9** to reach this level. Higher volume of suspension will not cause any problem.

Before taking the sample, it is necessary to mix the suspension well. In order to do that, start the agitation and leave it mix for 5 minutes. After this you may start the pump as well. When starting the pump, both suction **10** and exhaust **11**, **13** valves must be opened. After 5 minutes it is possible to take a suspension sample from valve **23.** First let flush the pipe and valve with some suspension collecting it into a bucket, then fill directly a graduated glass, so the level is flat at the top and no more suspension would fit inside. *Do not try to fill the glass only to the marked volume – the solids are settling and it is impossible to pour the suspension out without changing its properties.* After weighting the flask with suspension, fill it with water (the level must be exactly the same as it was with suspension) and weight it again. This allows calculating the density of the suspension from known mass and volume of the suspension:

$$\rho_S^0 = \frac{m_{PS}}{V_{PS}} = \frac{m_{PS}\,\rho_l}{m_{Pl}} \qquad (10)$$

where m_{PS} is weight of the suspension inside the glass and m_{Pl} is mass of water inside the glass. The initial weight fraction of the solids x_S^0 is then calculated by adding volumes of the solids and of the liquid:

$$\frac{1}{\rho_S{}^0} = \frac{x_S{}^0}{\rho_s} + \frac{1 - x_S{}^0}{\rho_l} \qquad (11)$$

where ρ_S^0 is density of the suspension, ρ_s solid phase density and ρ_l is density of water.

If the calculated weight fraction x_S^0 is smaller then value given for the task x_S, calculate the mass of solid material m_S which should be added into the tank using a mass balance of the solid material:

$$m_S^0 x_S^0 + m_s = \left(m_S^0 + m_S\right) x_S \qquad (12)$$

The mass of suspension is calculated using the suspension density determined before:

(13)

where V_S^0 is initial volume of the suspension in the accumulation tank. Any solid phase can be added only while the suspension is mixed and should be added in small amounts.

If the weight fraction of solid phase x_S^0 is higher then value given for the task x_S, calculate from the mass balance the mass of water m_l, which should be added before filtration:

$$m_S^0 x_S^0 = \left(m_S^0 + m_l\right) x_S \qquad (14)$$

In both cases repeat the measurement of the solids weight fraction during the filtration. Before any adjustments of the concentration in the tank, consult the action with your assistant.

IV. 2 Assembly of the filter and test of tightness.

Filtration is made on a single frame, which is to be inserted between the two plates. Then it should be covered by a pair of wet filtration membranes made of cloth. Both membranes have to be perfectly smooth and they have to be aligned with holes in the frame and plates. Also, the membranes have to cover the sealing surfaces of the plates and frame. Then the filter pressed is tightened by tightening a screw.

To check correct assembly of the filter, a tightness test has to be made – filter is filled with water and pressurized to filtration pressure. If the filter is well assembled, the water does not leak. Minor leakages – few droplets of water – will be sealed with the solid phase during the filtration. *The test has to be made when your assistant is present.* First switch of the pump. Then shut the filter outlet valves **17**, and valves into the tank **10**, **11** and **13**. Open the valve to the filter **15** and slightly open the pressurizing valve **12**. Remove air from the filter by opening the bleeding valve **21**. Then adjust the pressure to the test value 0,3 MPa and shut the valve **12**.

If the tightness test fails, the filter has to be opened and the filtration membranes refitted and the test repeated.

An industrial filter press contains higher number of frames and plates and the membranes are fitted over the plates, not the frames. In case of a single frame, is this procedure more suitable.

IV.3 Filtration

Filtration is carried out while constantly mixing the suspension in accumulation tank. The suspension is fed into the filter by a pump. Before starting, empty the measuring tanks **4** and close the outlet valves **19**. Valve **18** should be set to lead the collected filtrate into one of the tanks. For easier start of the measurement it is possible to set the liquid level in the measuring tanks to some distinct value – for instance 5 litres – so the liquid level is visible.

Start up of the filtration:

- fully open valve **13** (regulation of the pressure in the system),
- fully open filter outlet valves **17**,
- read the liquid level in both measuring tanks **4**.

The filtration, and thus the time and filtrate volume measurement, starts when the valve closing the pipe to filter **15** is opened. Precise reading of both is necessary for reliable evaluation of the filtration constants. The time is recorded for each 5 litre of the filtrate is obtained.

Prescribed filtration pressure is reached by slow closing the pressure regulation valve **13** while constantly watching the filtrate for clearness. Before some cake is produced, it is better to carry the filtration at small filtration pressure to reduce losses of the solid phase. The filtration pressure can be increased when the filtrate flowing from the filter is clear. During the rest of the filtration it is necessary to maintain the filtration pressure at constant value.

After first measuring tank is filled, the valve **18** is switched to reroute the filtrate into the second tank and while that one is being filled, empty the first (by opening valve **19**) preparing it for further measurement.

How long to filtrate? The filtration is finished, when the frame is filled with cake. For the filter in the students lab it is necessary to get a cake containing 5-6 kg of solid phase. Knowing the initial concentration of suspension, it is possible to calculate the filtrate volume from a mass balance of the filter. For reliable evaluation of the filtration constants some 15 readings of filtrate volume and time are necessary.

After finishing the filtration switch off the pump and then the agitation. Close the filter outlet valves **17**. When the suspension surface gets stable, read the volume of the suspension left in the accumulation tank.

IV.4 Removing and analysis of the cake

Loosen the screw holding the plates and frames together and move the back plate away. The cake is removed while still inside the frame and covered with the filtration membrane. Both the frame and the cake put on a balance and remove the frame in a way the cake will stay on the filtration membrane. Read the mass of the cake with the filtration membrane. Later (or before the filtration) weight also the wet filtration membrane.

Sampling the cake

To get an average sample of the cake, samples are taken from 9 places as is shown on the figure 2. The samples are taken into a baker of known mass.

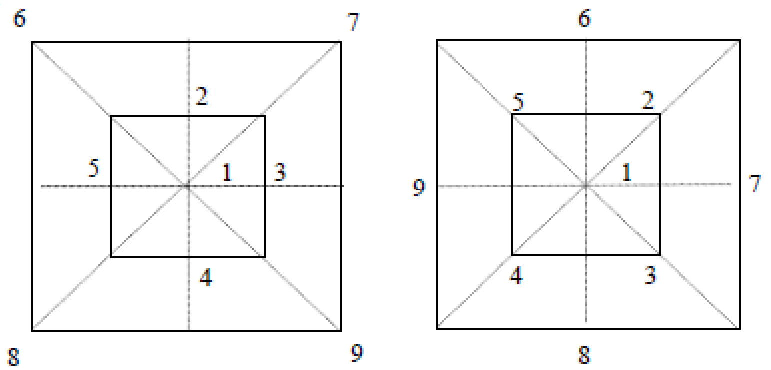


Figure 2. Diagram for sampling the filtration cake. The places from where the sample is taken are numbered from 1 to 9.

The baker with collected samples is weighted to find the sample mass m_{PK}. After this add enough water to get about 200 ml of suspension and mix it well. Transfer it carefully into a graduated glass, the volume of which is V_p = 250 ml. Fill the glass with water up to the mark and weight it. Subtracting the weight of the glass, the mass of the sample with water m_{PC} is obtained. The density of the filtration cake is then calculated from

$$\rho_K = \frac{m_{PK}}{V_{PK}} = \frac{m_{PK}}{V_P - V_{Pl}} \qquad (15)$$

where V_{PK} is volume of the cake sample, and V_{Pl} is the volume of water needed to fill the graduated glass with the cake sample, which is calculated

$$V_{Pl} = \frac{m_{PC} - m_{PK}}{\rho_l} \qquad (16)$$

where ρ_l is density of water. The weights should be known with 0.1 g precision.

Finally, the rest of the filtration cake is to put back into the accumulation tank.

IV.6 Cleaning

After finishing the experiment it is necessary to wash the filtration membranes and flush the equipment with water.

V Safety instructions

1. If it is necessary to release the pressure during the filtration, switch off the pump or open pressure regulating valve **14**.
2. Pay attention when assembling or disassembling the filter press. If your fingers might be jammed if they get into way. Rotate the filter screw slowly and pay attention that no one else is too close to its handle.
3. During the cleaning the water can not get into the pump drive.

VI Evaluation of experimental data

VI.1. Material balance and calculation of water content in cake

The aim of the material balance is to determine losses of the solid phase. The material balance is given by equations (1 – overall balance) and (2 – balance of the solid material). The weight fraction of the solid phase in the cake x_K is obtained from equation:

$$\frac{1}{\rho_K} = \frac{x_K}{\rho_s} + \frac{1 - x_K}{\rho_l} \tag{17}$$

where ρ_K is density of the filtration cake.

The calculations should be made with special attention to the precision, which determines the number of decimal places. For the material balance the least precise number is the volume of suspension, which is possible to read ±5litres. Thus it is enough to round the mass of the streams to whole kilograms (e.g. 401 kg). The weight fraction of solid phase in the suspension is always higher than 0.01, so the weight of the solid phase should be rounded to 1/10th of kilogram (e.g. 3.1 kg).

VI.2 Determination of filtration constants

Using experimental values of V_F calculate the quantity q_F from equation (3). The filtration area is for a filter press equal to twice the frame internal dimensions:

$$S_F = 2\, b\, h\, N \tag{18}$$

where *b* is the with of the frame, *h* is the height of the frame and *N* is the number of frames.

Pairs of values t_F and q_F are then fitted by quadratic equation (9) keeping the q_F as independent variable and t_F as dependent variable. If initial values of the constants a_0, a_1 and a_2 are needed, supply following values:

$a_0 = 1000$; $a_1 = 500$; $a_2 = 7.5\ 10^4$

The values of the filtration constants are then calculated from the values of the regression constants:

$$K_F = \frac{1}{2a_2} \tag{19}$$

$$q_M = K_F a_1 \tag{20}$$

A plot of experimental points and the fitted polynomial is an appendix to the report required by the “Aim” No. 3. *All experimental points have to be included in the graph but only those at constant filtration pressure should be used for the regression.*

VII Symbols

K_F	filtration constant (characterising cake resistance at filtration conditions)	m^2s^{-1}
q_F	filtration quantity, defined in (3)	m
q_M	filtration constant (relative resistance of the membrane and cake)	m
N	number of frames of the filter press	1
S_F	filtration area	m^2
v_F	rate of filtration, defined by (3)	$m\ s^{-1}$
α	specific hydraulic resistance of a porous material	m^{-2}
t_F	time of filtration	s
m	mass, see subscripts for detail	kg
ρ	density, see subscripts for detail	$kg.m^{-3}$

Subscripts

C	denotes total mass of graduated glass
F	denotes value for filtrate/ filtration
K	denotes value for cake
l	denotes value for water
P	denotes value for flask/glass
S	denotes value for suspension
t	denotes value for solid phase
Z	denotes value for losses
0,1	denotes value for start and end of the filtration period