UNIVERSITY OF SOUTH CAROLINA
CHEMICAL ENGINEERING LABORATORY

ECHE 460 LABORATORY PROCEDURE

Plate and Frame Filtration

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1. Objective and Background

Solid slurries are frequently encountered in chemical manufacturing operations. Filtration is a physical separation process that is used to isolate the solids as a "cake" from the liquid filtrate. The plate-and-frame filter is a common unit operation. It is inherently an unsteady state operation.

This experiment has been established to investigate the relationships existing between the rate of flow, pressure, and the thickness of the filter cake. Once these relationships are determined for a particular filter press, the size of the ancillary equipment (*i.e.* pumps and piping) can be determined. The apparatus in our laboratory can be operated in two modes; constant inlet pressure to the filter, or constant filtrate flow rate from the filter. Students will investigate both modes of operation experimentally. Finally, the knowledge gained will be used to design a plate-and-frame filter press operation.

2. Experimental Equipment

See the attached process schematic for details. It is important for you to study the apparatus and query your TA to gain full understanding. The slurry to be filtered is $CaCO_3$ (chalk for athletic fields) in water. The apparatus has the following main components:

1. Filter press and circulation pump.
2. Mixing tank and agitator.
3. O-Haus Model B100S 200-lb capacity scale, with Model I-10 indicator and computer for data acquisition.

In addition, the following items are used to determine the concentration of $CaCO_3$ in the slurry :

4. Specific gravity bottle.
5. Small scale for weighing specific gravity bottle.

3. Experimental Procedure

Caution: Do not place any plate elements on the expanded metal mesh (i.e., the screen) inside the recirculation tank. The screen cannot support the additional weight Any thing that falls into the tank could potentially destroy the impeller.

<u>Initial conditions (recirculation only)</u>

This mode is set up at the beginning of the laboratory period, or between runs, in order to pump the slurry continuously from the tank, through the pump, back to the tank. The filter press is bypassed when in recirculation mode.

Valve positions:	V-1 Full open V-2, V-3, V-4 full closed
Bench Scale:	Scale- zeroed with 28 gal. plastic container on scale

Computer: C:\labview\scale\scale.vi file opened and student's file entered in the file path block. Start the program running by pressing Control-R.

Slurry preparation and filter paper installation (while the system is in recirculation mode)

1. Remove the pressure transducer and clean any $CaCO_3$ from the piping.
2. Crank hand-wheel on the end of the filter press to loosen the sections of the press.
3. Install two sheets of filter paper between each section.
4. Crank hand-wheel to tighten the sections; maintain the numerical sequence for each section. Use the ratchet to torque the sections together.
5. Start mixing motor and filter press circulation pump to re-circulate slurry.
6. Use specific gravity bottle to measure slurry weight. Calculate weight fraction of $CaCO_3$ per attachment 1.

Operational procedure for constant pressure runs

1. Start the apparatus with mixer and filter press in circulation mode for several minutes.
2. Slowly open V-2 (filter press inlet valve).
3. Open V-3 (filtrate re-circulation valve) and slowly close V-1 (slurry re-circulation valve). This will start flow through the filter press. Remain in this configuration until the pressure increases to the desired value (the Teaching Assistant will provide these values) and the filtrate returning to the mixing tank is clear.
4. Once the filtrate is clear and the pressure is 1 psi above desired, open V-4 (filtrate outlet valve), close V-3 and start the computer data logging.
5. Maintain pressure at the desired value by gradually opening V-1.
6. Run filter press until the desired data has been obtained. Stop the flow of filtrate (by opening V-3 and closing V-4) when the desired pressure cannot be maintained, the filtrate is about to overflow the container, or the scale is reaching its maximum capacity (200 lb.). Stop data logging and computer program.
7. Fully open V-1, Close V-2 and V-3. Open V-4 to drain the water from the filter press.
8. Turn off the mixer and the filter press circulation pump. Return the filtrate and chalk to the mixing tank.
9. Repeat the experiment as many times as desired.
10. Clean area around filter press when experiment is completed.

Operational procedure for constant flow runs

1. Start Mixer and filter press in circulation mode.
2. Slowly open V-2 (filter press inlet valve).
3. Open V-3 (filtrate re-circulation valve) and slowly close V-1 (slurry re-circulation valve). This will start flow through the filter press. Remain in this configuration until the filtrate returning to the mixing tank is clear.
4. Once the filtrate is clear, open V-6 (filtrate outlet valve), close V-3 and start the computer data logging.

5. Maintain flow at the desired value (The Teaching Assistant will provide this value) by throttling flow meter inlet valve.
6. Run filter press until the desired data has been obtained. Stop the flow of filtrate (by opening V-3 and closing V-6) when the desired flow cannot be maintained, the filtrate is about to overflow the container, or the scale is reaching its maximum capacity (200 lb.). Stop data logging and computer program.
7. Fully open V-1, Close V-2 and V-3. Open V-4 to drain the water from the filter press.
8. Turn off the mixer and circulation pump. Return the filtrate and chalk to the mixing tank.
9. Repeat the experiment as many times as desired.
10. Clean the area around filter press when experiment is completed.

4. Theory

The governing equation for a filtration process has the familiar form of rate (filtrate flow rate Q) being directly proportional to a driving force and inversely proportional to a resistance.

$$Q = \frac{dV}{dt} = \frac{A\Delta p_f}{\mu R} \qquad (1)$$

or, expanding the total resistance R into its components

$$Q = \frac{dV}{dt} = \frac{A\Delta p_f}{\mu \cdot \left[R_f + R_c\right]} = \frac{A\Delta p_f}{\mu\left(R_f + \frac{\alpha c V}{A}\right)} \qquad (2)$$

where:

α = specific resistance of the cake (a constant, independent of cake thickness)
c = mass of solid/volume of liquid (m/L^3)
μ = viscosity of slurry (m/Lt)
Δp_f = pressure difference across the filter press (m/Lt^2)
A = filtering surface (L^2)
V = total volume of filtrate accumulated (L^3)
Q = rate of filtrate accumulation (L^3/t)
R_f = filter cloth resistance (an apparatus constant)
R_c = Cake resistance (increases with time as the thickness of the cake increases)
R = Total resistance, sum of filter cloth and cake resistances

The dimensions of the various quantities are given in mass m, length L, and time t. The students are responsible for performing and presenting all calculations in English units (lb_m, lb_f, seconds, and inches). The students must determine the appropriate units for the resistances. Unit consistency is always essential!

R_f is a constant, characteristic of the equipment and filter paper. For a given type of filter paper, method of assembly, etc. it is presumed that R_f is constant, independent of how the filtration process is run. α describes how much resistance to flow results per unit of solid processed per unit area. If one knows the concentration of solid in the slurry and the total amount of slurry processed, one knows the thickness of the filter cake. The overall resistance of the filter cake is R_c, and obviously R_c is a function of the specific resistance α and the amount of slurry processed. α is a constant for a given solid material but it may depend on filtration history (i.e. constant pressure or constant flow rate, amount of applied pressure, etc.) Both R_f and α must be evaluated from experimental data.

For ease of data analysis, the two commonly used conditions are constant pressure drop Δp_f and constant filtrate rate Q. Now equations (1) and (2) give the instantaneous flow rate versus the instantaneous pressure drop, and it should be noted that the cake resistance R_c also varies with time due to cake buildup. However, for constant pressure drop operation, Equation (2) can be integrated and rearranged into the linearized form shown below:

$$\frac{t}{V/A} = \frac{\mu}{\Delta p} R_f + \frac{\mu \alpha c}{2 \Delta p} \frac{V}{A} \qquad (3)$$

Equation (3) allows one to evaluate both R_f and α by linear regression of the data.

Similarly, a linearized equation for a constant filtration rate can be found by rearranging Equation (2)

$$\frac{\Delta p}{Q} = \frac{\Delta p}{V/t} = \frac{\mu}{A} R_f + \frac{\mu \alpha c}{A^2} V \qquad (4)$$

Both R_f and α can be found by regression of the data. Because each group will make several runs, it is of interest to compare the resistances under different conditions.

Because α depends on filtration conditions such as the pressure drop across the filter, this dependence should be investigated. The cake resistance (α) can be modeled with the Almy-Lewis equation

$$\alpha = k \Delta p^n \qquad (5)$$

where k and n are regression constants.

<u>Filter press design</u>

Experimental data are taken under either constant pressure drop or constant filtration rate conditions. In a real filtration operation, neither condition may be true. It is common to use the slurry pump to apply the maximum amount of pressure and flow through the filter press. In this case, Q will decrease with time while Δp_f will increase. It is desired to know how long it will take to process a given amount (say V = 5 or 6 cubic feet) of filtrate for the case where the pump is connected directly to the filter press. In other words, the throttle valve (V-2) is wide open and the bypass valve (V-1) is fully closed. This is a design calculation; do not run this experiment!

Once the constants R_f and α are determined for a particular filter press, the time it takes to process any given amount of filtrate can be calculated for a particular centrifugal pump. The characteristic pump curve for your pump is attached; for ease of computer calculations the characteristic curve should be fit to a quadratic polynomial:

$$\Delta p_p = a + Q(b + cQ) \tag{6}$$

where Q = flow through pump (assumed to be same as filtrate flow rate)
Δp_p = pump differential pressure
a,b,c = regression constants

For the configuration described above, we assume negligible resistance to flow between the pump and the filter press, therefore $\Delta p_p = \Delta p_f$. The amount of pressure head developed by the pump must be sufficient to overcome the pressure loss in the filter press. Therefore, at any instant of time, the operating conditions described by the pump characteristic curve (eq. 6) must match the operating conditions described by the instantaneous filter press design equation, eq. (2).

The design of the filter press requires solution of simultaneous equations and integration of the non-linear equation (2). This is an initial value problem. The equations will be solved numerically by a finite difference, forward-stepping integration method. The steps are summarized in the table below. The steps required to fill out the table are outlined below the table.

n	t	V_n	R_n	Q_n	$\Delta p_{f,n}$	Δt (fixed)	V_{n+1}
0	$t_0 = 0$	$V_0 = 0$	$R_0 = R_f$				V_1
1	nxΔt	V_1					

In the table, n is the step or increment number; beginning with n=0 this corresponds with time t=0 and cumulative filtrate volume V=0. These are the initial values. Now the total filtrate resistance R_0 at t=0 must be equal to the filter cloth resistance R_f, because no filtrate has flowed and no cake has accumulated ($R_{c,0} = 0$).

Now equations (2) and (6) must be solved simultaneously to give Q_0 and $\Delta p_{f,0}$. At this point, we have the instantaneous flow rate and pressure drop across the filter press at the initial moment when the filtration process is started.

Now comes an arbitrary decision. We will assume that the filter press maintains a constant Δp_f and flow rate Q for a short period of time Δt (say, 0.1 minutes?) If we rearranGe equation (2) to isolate dt, then differential time required to process an unknown amount of filtrate (from V_0 to V_1) is obtained by integration of eq.(2).

$$\Delta t = \int_{v_0}^{v_1} \frac{dV}{Q} \tag{7}$$

Now, analytical integration of equation (2) as suggested by equation (7) will result in a quadratic equation for V_1 and V_0 (or, for subsequent time steps, for V_{n+1} and V_n). Because V_n is known, equation (7) results in a single quadratic equation for the final unknown V_{n+1} in the table. Solve for V_{n+1}, put this in the next row in the table, and continue the computations until the desired amount of filtrate has been processed. This process is only approximate; accuracy can be improved by decreasing the size of the time step Δt.

5. Calculations and report requirements

Students should develop detailed objectives based on the team's specific results. The report should not necessarily be presented in the order given in this section.

1. Report all calculations in consistent English units.
2. Plot t/(V/A) versus V/A to obtain R_f and α for the constant pressure condition.
3. Plot Δp/Q versus V to obtain R_f and α for the constant flow condition.
4. Show how the linearized forms of the constant pressure and constant filtration rate equations were found.
5. Calculate the time necessary to process 190lbs. of filtrate using the attached pump characteristic curve (Note, our system uses two of these pumps in series). Compare with experimental time it takes to process 190 lbs. of filtrate.
6. Plot V, Δp and Q versus time for all runs and explain the curves.
7. Compare the resistances found for the constant pressure with the constant flow rate. Is there a difference between the two? Why or why not?
8. Determine the pressure dependence of α using the Almy-Lewis equation.

7. References

1. McCabe, W.L. and J.C. Smith, *Unit Operations of Chemical Engineering*, 3rd Edition, McGraw-Hill, 1976 pp. 932-942.
2. Coulson, J.M. and J.F. Richardson, *Chemical Engineering*, Vol. 2, Pergamon Press, 1960, pp. 414-421.
3. Walas, S.M, *Chemical Process Equipment Selection and Design*, Butterworth-Heinemann, 1990, pp. 305-334.

Attachment 1.

Calculation of weight fraction of calcium carbonate in water

$$\frac{1}{\rho_{tot}} = \sum \frac{x_i}{\rho_i}$$

$$\frac{1}{\rho_{tot}} = \frac{x_1}{\rho_1} + \frac{x_2}{\rho_2}$$

$$\frac{1}{\rho_{tot}} = \frac{\left(1 - x_2\right)}{\rho_1} + \frac{x_2}{\rho_2}$$

where

ρ_{tot} = slurry density

ρ_i = pure component density (either water or $CaCO_3$)

x_i = component weight fraction

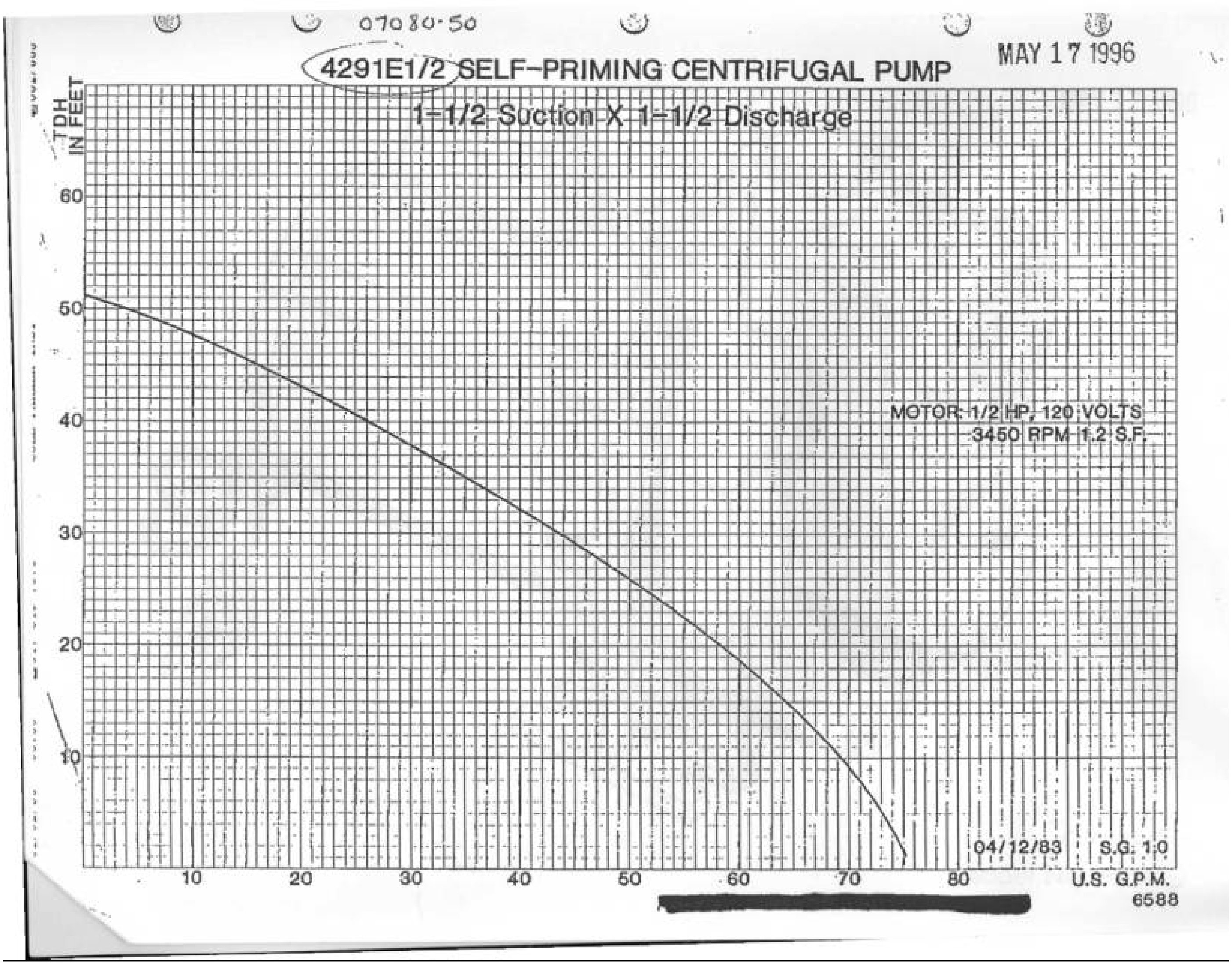

Figure A1. Pump curve for the two identical centrifugal pumps used in the filter press experiment.