Operating Instructions

Measurement of Moisture Content in Solids

Moisture content in solids needs to be measured in order to:

- Obtain porosity of cake produced by the filtration process (since pores of a wet cake are filled with water, the cake porosity corresponds to the volume fraction of water in the wet cake).
- Compute amount of partially wet solids needed for preparation of slurry of a specific concentration.

To obtain the moisture content in solids, perform the following steps:

1. Determine the tare weight of a sample collection pan.
2. Weigh a wet solids sample in the tared sample container.
3. Place the sample into the oven located in the fenced area outside the CHE building.

   **NOTE:**
   - The oven is very hot. Wear heat-resistant gloves when placing/removing samples to/from the oven.
   - Do not leave the gate to the fenced area open.
4. For measurement of the cake porosity, allow the sample to dry overnight.
   For estimation of moisture content of partially wet solids to be used in slurry preparation, it is sufficient to dry the solids for 15-20 minutes.
5. Weigh the dried sample in the tared sample container.
6. Calculate the moisture content.
7. Place the dried solids into a supply bin. Do not dispose of diatomaceous earth in the drain to prevent clogging of pipes.
8. Clean the sample containers and return them to their storage shelf.

Slurry Preparation

Usually, slurry for the batch filtration experiment is prepared from dry solids, since amount of solids needed for this experiment is relatively small and the necessary amount of dry solids is readily available. However, the continuous filtration experiment requires large amounts of solids, which are recycled from an earlier filtration experiment without drying them (due to the limited space in the oven). Hence, these solids are frequently partially wet.

It is recommended to work with slurries of concentration 10 wt% or less.

1. If you are using partially wet solids:
   a. Using a scoop, mix the partially wet solids in a bin for about a minute.
b. Obtain a small sample (about 30g) of the partially wet solids and estimate its moisture content using the method described above.

2. Estimate volume of the vessel (bucket for the batch filtration and slurry tank for the continuous filtration) in which you will be preparing slurry.

3. Calculate amounts of water and solids that you will need to mix in order to prepare slurry of a desired concentration. Remember to account for moisture content in solids. Make sure that the combined volumes of water and solids do not exceed 90% of the vessel volume.

4. Mix the solids and water in the vessel.

5. If you are using wet solids, verify that the prepared slurry has the desired concentration. This test is necessary because the moisture content of solids in the supply bin is not uniform. To measure the slurry concentration, take a slurry sample and measure its density. Use a dipper with a long handle to take slurry sample from the slurry mixing tank.

6. If necessary, add more solids or water to the slurry in order to achieve the desired concentration.
**Batch Filtration**

1. Prepare slurry following the instructions provided on the previous page.

2. Lock filter cloth in the filter frame with wingtip bolts and connect the filter assembly with the flask, as illustrated in Figure 3-1. Ensure that the filter cloth cover is securely in place between the cylinder and valve to prevent leakage. It is recommended to use a 4 L flask for the pressure-driven filtration and a 1 L flask for the gravity-driven filtration.

![Figure 3-1. Batch filtration system.](image)

3. Make sure that the valve below the filter is closed and have one group member stir the slurry in the bucket to prevent sedimentation of the solids. Pour the slurry into the graduated cylinder slowly ensuring that there is no solid left in the bucket.

4. For pressure-driven filtration, turn on the pump. Set the desired vacuum pressure using the vacuum regulator. After the pressure has reached a desired value, open the valve.

5. Measure dependence of the filtrate volume on time during the filtration process.

6. Drain the flask into the nearby grate and clean the filter frame.

7. Measure the cake porosity by weighing the wet and dry cake (see Measurement of Moisture Content in Solids).

8. Recycle the dried cake.

Note that the gravity-driven process is very slow and it becomes even slower as it progresses due to (i) cake build-up and (ii) reduction of the weight of the remaining slurry. Therefore, it is recommended to stop the gravity-driven experiment before all slurry passes through the filter but after sufficient amount of data was collected to obtain the cake permeability.
Continuous Filtration

Start-up procedure

1. Make sure that the feed valve (V1) and the drain valves (V5 and V6) are closed.
2. Make sure that the recirculation valve (V4) is open.
3. Open the cooling water valves:
   - Main cooling water valve (valve V2). This valve is located behind the filtrate tank as shown in Figure 3-2.
   - Check flowrate of the cooling water for the slurry pump. If it is not within the recommended range of 30-40 gallons per hour, adjust the needle valve V3 (see Figure 3-2).

It is vitally important to open the valves to let the cooling water flow, since the pumps will burn out without cooling water. Moreover, the vacuum pump has a flow switch that prevents pump from starting up if cooling water is not flowing.

4. Fill the slurry tank with water to the red fill line marked on the inside of the tank. Add the desired amount of solids to the slurry tank.
5. Turn on the master switch (see Figure 3-3).
6. Turn on the main power switch on the electric panel of the Continuous Filtration system (see Figure 3-4).

7. Plug in the stirrer power and turn on the stirrer switch (see Figure 3-4).
   **Be careful with the stirrer and do not put your hands into the tank while it is on.**

8. Turn on the slurry pump (see Figure 3-4). The recirculation valve V4 (see Figure 3-5) should be always open. Allow 5 minutes for mixing.

9. Turn on the rotary drum power (see Figure 3-4) and set the rotation speed. Recommended values are 1, 2, and 3 RPM.
10. Turn on the vacuum pump. If the pump does not turn on, check if the cooling water is flowing through the system. The vacuum pump has an automatic switch to prevent it from running without cooling water.

11. Slowly open the Feed valve V1 (see Figure 3-5) to avoid overflow of the filter basin.

12. Wait for the drum to make about half a turn so that the drum surface is covered by cake.

13. Use a combination of the Feed valve V1 and the pressure regulator shown in Figure 3-6 to set a vacuum pressure for the system.

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**During the Filter Operation**

1. After the vacuum pressure has reached a steady-state, wait for at least ½ of full rotation of the drum before starting data collection. This is necessary to ensure that the entire system has reached a steady-state.

The filter is now operational.
2. Collect the wet cake from the filter in a sample tray from the chute shown in Figure 3-7 and measure the cake production rate. Any extra cake should be collected in a bucket so that it can be recycled later.

Figure 3-7. Continuous drum filter. The circled area indicates location of the chute from which the solids are discharged after they are blown off of the filter surface.

3. Record the amount of filtrate collected in the tank as a function of time using the level indicator shown in Figure 3-8.

Figure 3-8. Filtrate tank.
4. When the filtrate tank is filled up\(^1\), it is necessary to pause the experiment and drain the tank:
   - Close the feed valve V1 (see Figure 3-5).
   - Turn off the vacuum pump and the drum motor. Keep the slurry pump and the stirrer running so that slurry remains well mixed while the tank is being drained.
   - Open the filtrate tank drain valve (V5), see Figure 3-8.
   - Close valve V5 after the filtrate tank is drained.
   - Restart the experiment and continue the run with the remaining slurry.
   - **Continue the experiment until all slurry is filtered. Do not drain unused slurry from the tank since this will clog the drain.**

5. When the slurry tank is close to empty, turn off the system as in step 4 and drain the filtrate tank. Do not turn the system back on yet.

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\(^1\) The volume of the filtrate tank is about 3 times smaller than that of the slurry tank.
6. Take the cover off of the filter and look behind the filter for the slurry return line shown in Figure 3-9. Use a hose to flush this pipe with water in order to remove any remaining solids from the pipe. The slurry return line is easily clogged due to the low pressure head, as well as several elbows in this pipe.

![Figure 3-9](image)

Figure 3-9. Top view of the filter and the filter basin. The entry into the recirculation line is circled. This line allows excess slurry to be returned to the slurry tank to prevent flooding.

7. Fill the slurry tank up with water up to the fill line and put all of the solids collected the previous run back into the slurry tank. This will preserve the desired concentration of slurry.

8. The system can now be started again with a full slurry tank.
Shut-down Procedure

1. Once the experiment is over, **clean the system**.
   - Flush the slurry return line, as described above.
   - Fill the slurry tank with water and run the filtration system without adding solids. This is necessary to flush remaining solids from the pipes and prevent clogging of the pipes.
   - Keep an eye on the filtrate tank to prevent overfilling. Drain when necessary.

2. Collect all solids in a bucket and add them back to the partially wet solids bin.

3. Close the feed valve V1.

4. Turn off the vacuum and slurry pumps, turn off the stirrer, unplug the stirrer power, and turn off the rotary drum power (see Figure 3-4).

5. Turn off the main power (Figure 3-4).

6. Turn off the master switch (see Figure 3-3).

7. Shut-off the cooling water.

8. Drain the filtrate tank and any remaining water from the slurry tank.

9. Use the hose to rinse off the entire system and floor (all equipment in the system is waterproof). Use squeegees to push any water on the floor into the grates nearby.