Chemical Engineering Laboratory
CHEG 4139

Caffeine Extraction

Objective:
The primary purpose of this experiment is to explore the different conditions that affect caffeine extraction and identify which variables have the greatest impact on caffeine extraction. Several variables may affect the amount of and rate caffeine is extracted from coffee grinds or beans, including (but not limited to) grind size, temperature, exposure to extract solvent, packaging, and residence time. A second objective for this lab is to allow students to practice experimental design. As such, this lab includes fewer detailed instructions than other labs. Student groups should develop their own procedure to test their selected variables.

Theory:
Caffeine can be isolated from coffee beans through a series of solid-liquid extraction and liquid-liquid extraction techniques. Many are familiar with the solid-liquid extraction through means of making coffee. The chemical most often associated with coffee, caffeine, can by further isolated/purified from coffee through liquid-liquid extractions.

As the liquid passes through the bed of coffee grinds, the caffeine moves from the solid phase to the liquid phase. Through the use of several assumptions, these results can allow the calculation of an effective mass transfer coefficient. There are several approaches to calculate this and developing the approach is the responsibility of each team.

Caffeine is more soluble in dichloromethane than in water. Consequently, the addition of dichloromethane to a coffee solution containing caffeine will result in the transfer of caffeine to the dichloromethane solvent. The solvent containing caffeine can be separated from the coffee solution with a separatory funnel. The concentration of caffeine can roughly be determined using a calibration curve and a reusable quartz cuvette. The amount of caffeine extracted, however, may be dependent on the chosen methods.

Safety Precautions:
1. Dichloromethane is a hazardous chemical. Review the SDS.
2. Dichloromethane should only be opened in the hood. Wear all PPE when handling dichloromethane. *Silver Shield gloves will be provided to students who will work with dichloromethane extractions. These may be reused by the same students until the experiment has concluded.*
3. If dichloromethane comes in contact with skin, ri nse with soap and water for 15 minutes.
4. If dichloromethane comes in contact with eyes, rinse for at least 15 minutes.
5. All waste containing dichloromethane should be disposed of in the appropriate hazardous waste container.

Available Variables: Examples of variables include: size of the coffee grinds, temperature, flow rates, mass of coffee, separatory funnel inversions, size of column, coffee brand, column packing method, and additional variables selected by the group.

Available Materials: Additional materials may be available. Confirm with your teaching assistant.

Dark Roast Coffee Beans	Medium Roast Coffee Beans	Espresso Coffee Beans
Column (CG-1186-06)*	Column (CG-11856-10)*	Burr Grinder
Electric Grinder	Cotton Balls	Sand
Spectrophotomer	Reusable Quartz Cuvette	Methylene Chloride
Silver Shield Gloves	125 mL Separatory Funnel	Face Shield
Stir/Hot Plate	Digital Thermometer	Ring Stands
Clamps	Flowmeter	Gear Pump Drive
Tubing	Balance	Calipers

*Glassware from ChemGlass. Product details available through their online catalog.

Figure 1. Solid-Liquid Extraction Set Up. Hot water bath and flowmeter connected by a gear pump drive.

Pro Tips:

1. To expedite sample collection, mark 15 mL on the Erlenmeyer flasks prior to starting sample collection.
2. It takes a good amount of time for the feed tank contents to heat to the desired temperature. Fill the tank and start heating as soon as possible.

3. Consider how many variables to collect to allow for adequate statistical analysis (e.g. mean, standard deviation, t-tests).

General Experimental Procedures:
The following procedures apply to specific elements of this lab but it is up to each team to build upon these.

Calibration Curve Development

1. Create a calibration curve using a starting solution with a known caffeine concentration. Caffeine concentrations should be in a dichloromethane solvent.
    a. You will be given a pure caffeine sample with known weight.
    b. Add methylene chloride solvent to the sample to create a highly concentrated solution.
    c. The highest concentration of solution that you may include in your calibration curve will be roughly 2.60 mg to 2.80 mg in 100 mL of methylene chloride.
2. Calibrate the flowmeter.

Solid-Liquid Extraction

3. Fill the feed tank with DI water and heat to the desired temperature.
4. Grind coffee beans in grinder.
5. Pack the column
    a. Use a small piece of cotton to plug the bottom of the column
    b. If available, you may use sand to give the stationary phase an even base
    c. There are a number of ways to pack a column, choose a method and explain your choices
6. Turn on the pump and set the desired flowrate.
7. As the water level approaches the mark, adjust the outlet valve so the water level stays constant (i.e. reaches steady state).

Liquid-Liquid Extraction

8. Pick a point from which to time the run and stay consistent with the timing system.
9. Collect 15 mL samples from the outlet stream at predetermined time intervals in pre-labeled Erlenmeyer flasks.
10. When ready for extraction, add the 15 mL sample to the separatory funnel.
11. Next, add 15 mL of dichloromethane to the separatory funnel. Do not shake.
12. Place the stopper on top of the funnel and invert a desired amount of times. Release any built-up pressure. Allow the liquids to settle and separate into two phases.
13. Remove the dichloromethane phase into a new flask or bottle.
14. Repeat steps 13-15 twice more with the original sample using 15 mL of fresh dichloromethane each time. Collect the dichloromethane extracts from the three extractions in the same bottle and measure its volume.
15. Transfer a portion of the dichloromethane extract into the quartz cuvette and place it in the spectrophotometer. Take a reading, and record the absorbance at 275 nm.

It has been determined by previous students that three extractions are enough to extract all the caffeine from the 15mL sample. Feel free to conduct your own investigation to prove them right or wrong!

Calibrating the Flowmeters

1. Set the desired flowrate using the pump knobs
    a. Make sure to read the flowmeter float at its widest point
2. Ensure the temperature is at the setpoint
3. Allow the system to equilibrate by running the system for the equivalent of one residence time
4. Label and weigh 2-4 50-mL Erlenmeyer flasks; these will be the liquid collection vessels
    a. Weigh the collected liquid and record the weight
    b. Return the collected liquid back to its container
5. Perform step 4 two more times for a total of three data points
6. Repeat steps 1-5 for three to five different flowrates