Chemical Engineering Laboratory
CHEG 4137/4139

# 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.

**Theory:**
Extraction is a technique used to isolate a certain compound from a liquid solution. Caffeine is more soluble in dichloromethane than in water; adding dichloromethane to a coffee solution containing caffeine will result in the transfer of caffeine to the dichloromethane solvent. This solvent is then separated from the coffee solution using a separatory funnel and the concentration of caffeine is determined using a calibration curve and a reusable quartz cuvette.

**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, rinse 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:** Size of coffee grinds, temperature, flow rates, mass of coffee, tea bag selection, separatory funnel inversions

**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.

**Experimental Procedure:**
1. Create a calibration curve using solutions of known caffeine concentrations. Caffeine concentrations should be in a dichloromethane solvent.
2. Calibrate the flowmeter.
3. Fill the feed tank with DI water and heat to the desired temperature.
4. Grind coffee beans in grinder.
5. Fill an empty tea bag with the desired weight of ground coffee and staple the bag shut.

6. Place the bag in the reactor and add a predetermined amount of DI water. This will be your reactor volume. Mark the level in the reactor to be used as a frame of reference for the rest of the experiment.
7. Discard the bag and water, then prepare a new bag to be used in the experiment. Ensure the outlet valve connected to the reactor is closed.
8. Turn on the pump and set the desired flowrate.
9. As the water level approaches the mark, adjust the outlet valve so the water level stays constant.
10. Pick a point from which to time the run and stay consistent with the timing system. Add the coffee-filled bag to the reactor.
11. Collect 15 mL samples from the outlet stream at predetermined time intervals in pre-labeled Erlenmeyer flasks.
12. When ready for extraction, add the 15 mL sample to the separatory funnel.
13. Next, add 15 mL of dichloromethane to the separatory funnel. Do not shake.
14. 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.
15. Remove the dichloromethane phase into a new flask or bottle.
16. 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 <u>same bottle</u> and measure its volume.
17. 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!

**Calibrate 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