

# Corn mash and distillation

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|------------------------|--|
| <b>Focus questions</b> | How can we model the production of ethanol on a small scale?   |
| <b>Vocabulary</b>      | Starch, glucose, fermentation, anaerobic respiration, ethanol, coproducts, distillers grains, corn oil, carbon dioxide |

In this lesson you will create a corn mash that will undergo the fermentation process to prepare for distillation. Next, you will separate the **distillers grains** from the **filtrate** and distill ethanol from the filtrate. You will be asked to construct and revise an explanation for the use of corn in commercial ethanol production as you use anaerobic respiration and distillation processes to make ethanol. You may need to do additional research to propose future solutions to improve the efficiency of ethanol production.

## Materials

- Hot plate
- 110V heating mantle
- 100 or 1000 mL distillation apparatus
- Condenser tube
- Dial thermometer
- Graduated cylinders (10, 100 mL)
- Large watch glass covers
- Beakers (100, 250, 600, 1000 mL)
- Deionized water
- Hammered dent corn
- Scale or triple beam balance
- Glass vials with caps (or a small beaker)
- Buffer solution (pH 5)
- Yeast solution (20 g yeast/100 mL water,
- 49–55° C)
- Amylase solution (3 tsp/100 mL water)
- Glucoamylase solution (3 tsp/100 mL water)
- Funnel
- Thermal gloves
- Glass stir rod
- Cheesecloth/plastic sieve
- Safety glasses
- Hot gloves
- Optional: pipette pump
- Optional: 10 mL serological pipettes
- Optional: aluminum foil
- Optional: paper towels

## Procedure

### Day 1: Preparation of corn mash

1. Bring 300 ml of water to a boil in a 1000 ml beaker
2. Weigh out 100 g of hammered dent corn (or corn flour) and add to boiling water.
3. Record observations in the Data section. Stir well. Bring the mixture to a boil. Reduce temperature to maintain gentle boil. Stir constantly for 15 minutes. **Be careful not to let it burn.** (If the mixture becomes too dry, more water may be added).
4. Remove the beaker from the hot plate and allow it to cool to touch (35-40 °C or 95-104 °F). Record observations on consistency, color, and smell in Data section.

5. While waiting for corn mash to cool:
  - a. Measure 100 mL of distilled water and pour into a 250 mL beaker.
  - b. Shake the **amylase solution** well.
  - c. Measure 10 mL of the **amylase solution** into a small graduated cylinder and add to the 250 mL beaker of water and stir.
6. After corn mash has cooled to temperature above,
  - a. Add the amylase and water mixture to the corn mash and stir to evenly incorporate.
  - b. Let sit for 10 minutes, stirring occasionally with a glass rod.
7. At the end of the 10-minute period,
  - a. Shake the **buffer solution** and **glucoamylase solution** well.
  - b. In a 100 mL beaker, mix 35 mL of the **buffer solution** (to maintain a slightly acidic pH), 10 mL of **glucoamylase solution**, and 10 mL of the **yeast solution** together.
  - c. Pour the mixture into the corn mash. Stir the entire mixture well.
8. Place a piece of plastic wrap over the mouth of the beaker and label. Fermentation will occur so do not secure it too tightly.
9. Place your beaker on the counter and allow it to sit for **3 days** so that the enzymes have time to work. Stir the corn mash each day to maximize fermentation. *Optional: Place corn mash in an incubator set at 32 °C/90°F (optimal temperature for yeast metabolism).*

#### **Day 4: Distillation of ethanol from corn mash**

1. Set up the distillation apparatus as demonstrated by your instructor. (Before you use your distillation apparatus, have your lab instructor inspect it).
  - a. Make sure to either grease or wet the ground glass joints before connecting them. This helps to prevent any vapor from escaping the joints and to keep the joints from freezing together.
  - b. Filter out your distillers grains by using cheesecloth or coffee filters. Be sure to press on the mash to filter out all of the liquid. Pour about 50–75 mL of your solution in the 100 mL distilling flask so that it is a little more than half full.
    - *If you are using a large distillation flask (1000 mL), put the entire class' solution into one flask.*
    - *If necessary, add enough distilled water to the flask to reach the halfway point of the flask to ensure even heating of distillate.*
  - c. You will use a heating mantle to provide the heat necessary for the distillation. *\*Do not use a bunsen burner!* The inside of the mantle will eventually become extremely hot. In order to control the heat, you can raise or lower the temperature of the heating mantle. Make sure the distillation flask and condenser tube are properly supported! The outside of the mantle is relatively cool to touch.
2. The best separation of alcohol will occur if the distillation is done slowly. Ethanol's boiling point is 173.1°F/78.37°C and water's is 212°F/100°C— be sure to keep the temperature between these two boiling points. Collect the ethanol distillate samples in a small beaker to be used for the alcohol flame test. Pour the distillate samples into a 4 dram capped vial for storage until ready to do the flame test.
3. When you are no longer able to distill your product, turn the heating mantle off and allow the distillation apparatus to cool before disconnecting and cleaning.

**Table 1: Data**

| Day 1                          | Consistency | Color | Smell |
|--------------------------------|-------------|-------|-------|
| Before boiling (step 2)        |             |       |       |
| After boiling (step 3)         |             |       |       |
| After enzyme addition (step 4) |             |       |       |

| Day 4                             | Consistency | Color | Smell |
|-----------------------------------|-------------|-------|-------|
| Before distillation (step 1)      |             |       |       |
| First distillate sample (step 3)  |             |       |       |
| Second distillate sample (step 4) |             |       |       |

**Table 2: Flame test**

|          | Time | Flame properties |
|----------|------|------------------|
| Sample 1 |      |                  |
| Sample 2 |      |                  |

**Alcohol flame test**

1. *This test should only be done in a fume hood after the ethanol distillation is complete and there is no vapor in the air.* You will be testing the ethanol distillate for alcohol concentration by lighting it on fire. The longer the flame burns, the greater the alcohol concentration. If the distillate does not burn, the water concentration is too high (over 50%).
2. Place a watch glass in the fume hood and pour 2 mL of your ethanol distillate on it.
3. Turn the fume hood on and lower the window, light your distillate, record the time the flame burns, and observe.
4. Measure the amount of remaining water on the watch glass and calculate the alcohol percentage of your product.

**Salt wash (optional)**

Students can separate remaining water from the ethanol distillate by adding potassium carbonate,  $K_2CO_3$ , which is soluble in water but not in ethanol. The  $K_2CO_3$  and water will form an alkaline solution and separate from the ethanol to form a dense, bottom layer with the ethanol remaining in the top layer.

## Reflection

1. What effect does the physical heating have on the corn mash?
2. Explain how each enzyme (amylase and glucoamylase) change the corn mash mixture in preparation for fermentation.
3. What is the function of the yeast during the fermentation process? How did the consistency of the corn mash change during the 3 day fermentation process?
4. What are other ways you can experiment to make the fermentation process more efficient?
5. Describe the physical changes that your corn went through during its transformation into ethanol.
6. What byproducts result from ethanol production?
7. What are efficient and economical uses for the coproducts (carbon dioxide and distillers grains) that are generated during ethanol production?
8. Construct an explanation for using corn in the process of commercial ethanol production based on evidence from student investigations and design logical solutions for the coproducts that are produced.

## Rubric for self-assessment

| Skill  | Yes | No | Unsure |
|--|-----|----|--------|
| I generated data and constructed an explanation for the use of corn in commercial ethanol production.                                  |     |    |        |
| I suggested viable solutions for the industrial application of the remaining coproducts produced in the commercial ethanol production. |     |    |        |