

# CORN WET MILLING LAB

Eric Olson, Indian Hills Community College, Ottumwa, IA  
Modified by Noreen Warren, Madison Area Technical College

## THINK SAFETY, WEAR YOUR SAFETY GLASSES!!!

### Purpose

The United States produces 9.3 billion bushels of corn each year, most of it in the Midwest corn belt. Most students believe that all the corn that is grown feeds farm animals or people at the annual picnic, and a good amount of it does, but what happens to the corn not consumed by cattle, pigs or people? A large amount of corn never makes it to the dinner table. Corn Wet Milling is an industrial process that converts corn to a wide variety of by-products. The wet milling industry is the largest non-feed user of corn, using approximately 1 billion bushels annually. This lab looks at the separation steps in the industrial processing of corn.

### Background

Corn wet milling is a complex industrial process. Large wet millers process approximately 100,000 bushels of corn daily, and the primary product is starch and starch derived chemicals. This cornstarch is then processed and used as food and industrial products. It is routinely used as an adhesive, for manufacture of papers, and as filler for pharmaceuticals. It can be converted into an enormous assortment of industrial chemicals now produced from petroleum sources. Most plastics used in the U.S. are made from materials that come from petroleum. Petroleum products are a major problem in our rapidly filling landfills. New biodegradable plastic products are being made from corn such as garbage bags, car parts, and packing peanuts. By simply combining about 6 percent cornstarch with the plastic, the new corn plastics will degrade in less than two years! Chemists say there's nothing now made from petroleum (non-degradable) that can't be produced from biodegradable corn.

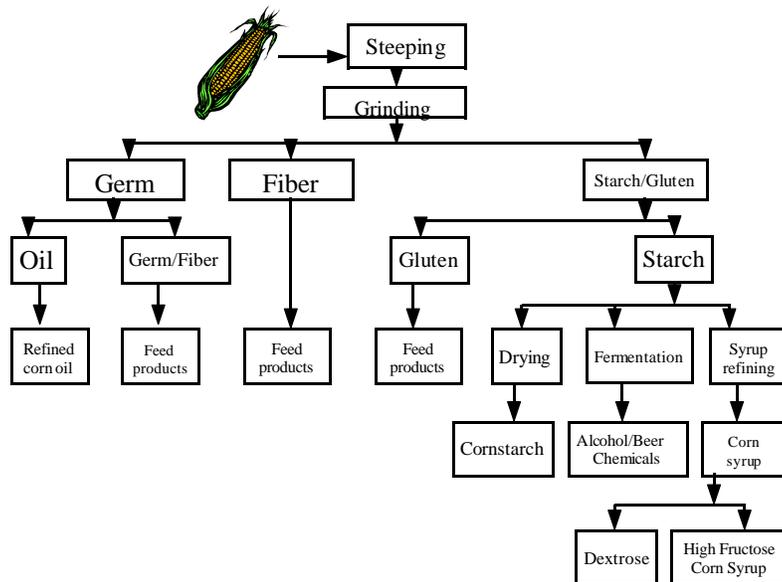
In addition, cornstarch derived from wet milling can be fermented to produce alcohol. The distilling and brewing industries both use cornstarch as the raw materials in their fermentation processes. It is a raw material for the production of energy. Ethanol is a high performance fuel made from cornstarch. It's safe for the environment, reducing air pollutants by more than 50 percent because it burns much cleaner than gasoline.

Other products obtained from this process are corn oil, bran and protein. The oil is further processed to produce various salad oils and similar products. The protein and bran are used primarily as feed ingredients. Additional processing produces modified starches, maltodextrans (slightly degraded starch), sweeteners, and other chemicals/pharmaceuticals derived from fermentation.

### The Wet Milling Process in Industry

The Wet Milling process starts with dried corn kernels that are inspected and cleaned to remove the cobs, chaff and other debris. The corn is then soaked in large tanks with small amounts of sulfur dioxide and lactic acid. These two chemicals, in water held at 122°F (50°C), help soften the corn kernel over a 24-48 hour steeping period. During this time, the corn swells and softens and the mild acid conditions loosen the gluten bonds to release the starch. After steeping, the corn is coarsely ground.

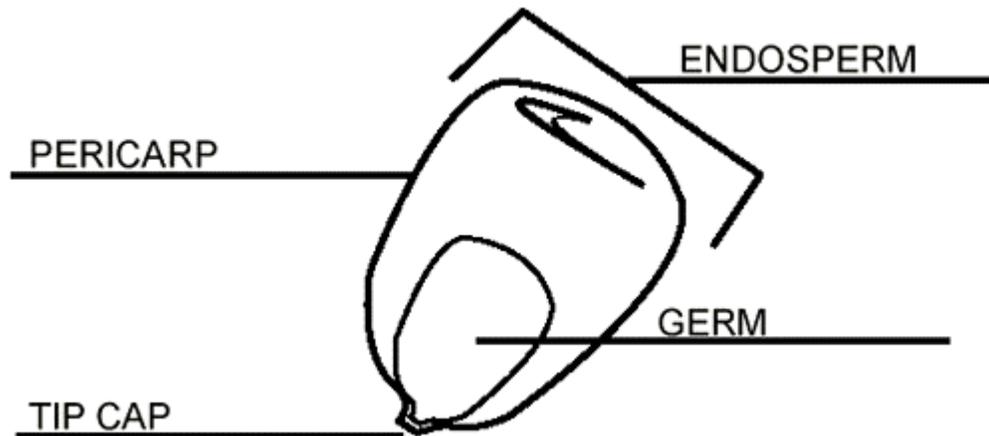
The ground corn and some steep water are passed through a separator, which essentially allows the germ, or the lightweight oil-containing portion, to float to the top of the mixture and be removed. The fibrous material is screened off, and the starch and protein are separated by density using large centrifuges.



Uses for corn in the wet milling process

## The Basic Biology of Corn

Corn is grown industrially for its resource rich kernel. There are four major parts of the corn kernel. The seed coat (pericarp), germ (embryo), the tip cap and the endosperm. The seed coat is a layer of fiber that protects the kernel from attack by microorganisms and insects. The germ is the only living part and contains a miniature plant with preformed leaves, a radicle root and an oil rich cotyledon. About 25% of the weight of the germ is oil. The tip cap is the point where the kernel is attached to the cob. It is the major entry for food and water into the kernel. The endosperm comprises about 80% of the kernel and is composed primarily of starch (or sugar in sweet corn).



Industrial separation of the useful components of corn takes advantage of the biology of the corn kernel. The oil is in the germ, while the starch and gluten are found in the endosperm. Starch is a complex carbohydrate consisting of linked glucose molecules. Gluten is a protein found in corn and other grains. The chaff or fibrous portions of the corn kernel are in the remains and are used for animal feed. A method to isolate the economically important chemicals from corn utilizes the compartmentalization that is found in the corn kernel.

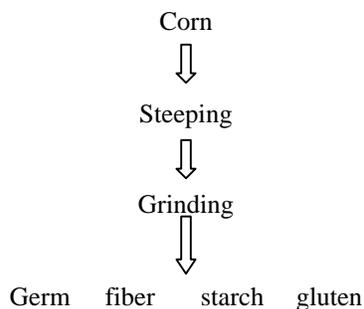
## Corn Varieties used for Wet Milling

There is an economic advantage to find the best variety of corn kernel to be used in the wet milling process. Developers of new corn varieties are constantly trying to find the optimum characteristics for use in this industrial process. The only clear corn kernel characteristics that result in higher yields of starch for wet millers are large kernels that are softer (i.e., more floury) in texture. Softer kernels probably result in higher starch yields because the starch is less tightly "cemented" together (by protein) within the kernel, so it is easier to separate from the protein and other kernel components. It is important to characterize corn varieties for their productivity in the wet milling process. While an easy method to predict wet-milling quality has yet to be fully developed, there are clear differences in wet milling yields found between corn hybrids, and within a single hybrid grown under different environmental and cultural practices. Corn hybrids have already been developed that are optimized for different growing regions, but now to expand markets, corn must be "genetically customized" for its eventual use, whether it be for feed or industrial use. The research to find out what corn properties are most desirable for particular end uses is just beginning. This lab will compare different genetic varieties of corn to determine which are optimal for starch isolation.

Yellow dent corn is commonly used in industrial corn wet milling. It has a yield of 61% starch, 16% gluten, 20% germ and 3% fiber. The high percentage of starch in this variety of corn makes it ideal for use in the production of cornstarch as the material for further processing to sweeteners, alcohol fermentation, the construction industry and other uses. Other varieties of industrially important corn include high oil corn for the production of corn oil and high amylose corn for the production of a specific usage starch.

## Synopsis

The student will weigh, steep, grind, and separate components from a pre-determined amount of corn. The student will then determine whether the recovery is within accepted specifications (+/- 5%) of theoretical totals. The starch can be saved for further use.



## MATERIALS

yellow field corn (yellow dent #2) obtain from a farm supply house or birdseed company (corn on the cob for squirrel feed) if possible find other types or try sweet corn

lactic acid (dl-lactic acid 85% syrup Sigma # L1893)

sodium bisulfate (Sigma #9000)

NaCl

tert-amyl alcohol (Fisher #A730-1 or Sigma # A1685)

distilled or purified water

erlenmeyer flask (500mL)

beakers

pipettes and pipette bulbs

weigh boats

analytical scale

incubator (shaking at 54° C)

corn grinder (made from an ordinary household blender) See note at end

kitchen sieve

## SOLUTIONS

### Steep Water

|                             |         |
|-----------------------------|---------|
| 2% v/v dl-lactic acid syrup | 20 mL/L |
| 0.2% sodium bisulfate       | 2 g/L   |

### 2 M NaCl

NaCl 116.9 g/L

tert-amyl-alcohol: water (4:1)

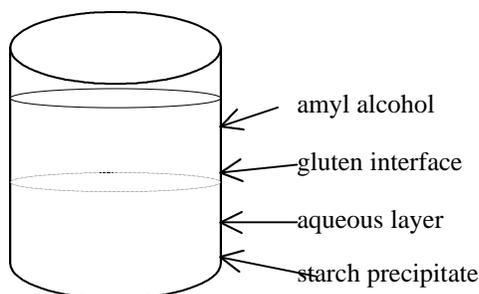
## BASIC PROCEDURE

Day 1 (time: 30 minutes)

1. Count and weigh 44 kernels of corn for each variety tested
2. Place corn in 500 mL Erlenmeyer flask.
3. Add approximately 250 mL of Steep Water.
4. Incubate with slow shaking (200-250 rpm) at 54° C
5. Incubate for 45-48 hours

Day 3 (time: 2 hours)

6. Drain off the steep water.
7. Rinse corn with tap water.
8. Place corn in grinder with approx. 60 mL of distilled water.
9. Grind corn for approx. 15 sec. (medium setting) or until whole, intact germ can be seen. Rinse down the sides of the grinder with small amounts of additional distilled water to insure proper degree of grinding.
10. Pour the contents of the grinder through the sieve catching the permeate in a beaker, additional rinsing of the grinder may be necessary. (The permeate fraction contains the emulsified starch/gluten)
11. Place the contents of the sieve into the 2M NaCl, the corn germ will float on the surface.
12. Collect as much of the corn germ and transfer to a pre weighed weigh boat to dry.
13. Pour the 2M NaCl and the remainder of the ground corn into the sieve. (Retain the 2M NaCl flow through to use a second time)
14. Place the contents of the sieve (fiber/germ and starch/gluten chunks) and the emulsified starch/gluten permeate (step 10) back into the blender and grind for approx. 15 sec. (med. setting), again some rinsing may be necessary to insure proper mixing.
15. Repeat steps 11-13, until there is little or no germ that can be separated by floating the mixture over 2M NaCl.
16. The retentate in the sieve should be mostly fiber, place in a pre weighed weigh boat to dry.
17. Pour the starch/gluten permeate into the amyl alcohol/ water solution. The starch will settle on the bottom and the gluten will settle in the alcohol/water interface.
18. Allow this to settle out for approx. 1 hour.
19. Using the pipette bulb and a large pipette, siphon off the amyl alcohol into a collection container (save for future use) until the yellow gluten level is reached.
20. Siphon off the gluten layer and place in a pre weighed weigh boat to dry.
21. Siphon off the water layer until the starch layer is reached and place in a pre weighed weight boast to dry.
22. Allow the products to dry for several days.



Day 4 (time: 30 minutes plus 60 minutes for calculations and write-up)

- 23 Weigh the dried products. Compute the percent of the total weight of all the components. Determine the deviation from the theoretical yield and calculate the percent deviation.

Theoretical yield for yellow dent corn:

Starch = 61% of total weight  
 Fiber = 3 %  
 Gluten = 16%  
 Germ = 20%

|                    | weight | actual % of total | theoretical - actual % | % deviation |
|--------------------|--------|-------------------|------------------------|-------------|
| whole corn kernels |        |                   |                        |             |
| germ fraction      |        |                   |                        |             |
| fiber fraction     |        |                   |                        |             |
| starch fraction    |        |                   |                        |             |
| gluten fraction    |        |                   |                        |             |
| total              |        |                   |                        |             |

Did you retrieve all of the original corn weight? Why or why not?  
 Could there have been protein or weight lost in the steep water? Why or why not?  
 Could the NaCl have altered your weight results? Why or why not?  
 If your results are off, what do you think could have caused this?  
 Is there a way that this exercise could have been done better? How? Why?  
 If a corn crop yields 150 bushels/acre has 8% protein what is the protein yield?

$$150\text{bu/acre} \times 56 \text{ lb./Bu} \times 0.08 = 672 \text{ lb./acre}$$

Compare the varieties of corn tested. What would be the advantages and disadvantages of each type of corn for industrial purposes.

## References

Corn Quality for Industrial Uses (<http://www.unl.edu/pubs/g1115.htm>) Published by Cooperative Extension University of Nebraska-Lincoln.

Corn the Great American Crop (<http://www.agry.purdue.edu/ext/corn/pubs/agry9301.htm>) Dr. RL Nielson Agronomy Department Purdue University.

Uses of Corn (<http://www.kycorn.org/cornuses.html>)  
 Corn Refiners Association (<http://www.corn.org>)  
 Crop Insights ([http://www.pioneer.com/usa/crop\\_management/national/corn\\_grain\\_protein\\_99.htm](http://www.pioneer.com/usa/crop_management/national/corn_grain_protein_99.htm))  
 Pioneer Hi-Bred International Crop Management Research and Technology.

Watson Stanley A., and Paul E. Ramstad, eds., *Corn: Chemistry and Technology*, American Association of Cereal Chemists, Inc., St. Paul Minnesota, USA, 1987.

## Additional activities

Determine the amount of protein in each of the fractions  
 Determine the amount of starch in each of the fractions  
 Determine the % starch/protein in each of the fractions. (See attached exercise)  
 The starch fraction is further processed in industry for the fermentation (beer and distilled alcohol)  
 industries devise a lab to test alcohol production with the starch fraction.

## Teacher's notes

The Grinder used is an inexpensive Hamilton Beach or equivalent purchased at a discount store for about \$10.00. Find some help and have the blades ground flat. It is important that the blades are flattened or dulled, you do not want a chopping action rather a grinding or blunt force action.

The cornstarch can be later used in the STARCH HYDROLYSIS lab (attached) or an ethanol fermentation lab. This mimics the processes in industry.

## GLOSSARY

|              |   |
|--------------|---|
| chaff        | Fibrous debris such as bracts removed before processing.  |
| cotyledon    | Part of the embryo of a seed plant, that enlarges upon germination, and becomes the first green leaves.                                       |
| embryo       | The rudimentary plant that is enclosed within the seed.   |
| endosperm    | The nutritive tissue within a seed that feeds and surrounds the embryo. The endosperm is the storage place for starch.                        |
| germ         | The embryo in the seed which develops into the growing plant.   |
| gluten       | The protein fraction of some cereal grains that can be isolated because of its density.<br>Gluten is lighter than water.                      |
| kernel       | The grain or seed enclosed within a husk.   |
| pericarp     | The outer covering of the kernel that protects it from deterioration; the seed coat.  |
| permeate     | The fraction of a solution that is capable of flowing through a filter.   |
| radicle root | The part of a plant embryo that develops into a root.   |
| retentate    | The fraction of a solution that is held on the surface of the filter.   |
| seed coat    | The outer protective covering of a seed, the pericarp.  |
| starch       | A complex carbohydrate found in vegetables and grains that is composed of linked glucose molecules.   |
| steeping     | To soak in liquid to soften and extract components.   |
| tip cap      | The attachment point of the kernel to the cob.  |
| wet milling  | Industrial process that isolates the industrially important fractions of the corn kernel by nature of their partition within the corn kernel. |