National Corn-to-Ethanol Research Center: Commercialization Research for the Fuel-Ethanol Industry

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National Corn-to-Ethanol Research Center

- Conducts industry- and government-sponsored research to improve the production of ethanol from grain-based feedstocks
- Provides third party validation, commercial testing, and assistance in developing new technologies
National Corn-to-Ethanol Research Center

pilot plant
National Corn-to-Ethanol Research Center

pilot plant

analytical lab
National Corn-to-Ethanol Research Center

pilot plant

fermentation lab

analytical lab
National Corn-to-Ethanol Research Center

- fermentation lab
- pilot plant
- analytical lab
- workforce training
NCERC Mission

Facilitate the commercialization of new technologies to produce fuel ethanol more effectively, such as

- technologies that can improve the efficiency of ethanol production from corn and other starch-based feedstocks
- technologies that will improve the reliability of ethanol production processes
- technologies that will increase the value of coproducts of the fuel-ethanol industry
Commercialization of New Technologies to Improve Fuel-Ethanol Production

1 bushel of corn (56 lbs.) + yeast + amylase enzymes + 92,600 Btu + 12 gal water = 2.7 gallons (18 lbs.) ethanol + 18 lbs. CO₂ + 18 lbs. DDGS
Commercialization of New Technologies to Improve Fuel-Ethanol Production

1 bushel of corn (56 lbs.) + yeast + amylase enzymes + 92,600 Btu + 12 gal water = 2.7 gallons (18 lbs.) ethanol + 18 lbs. CO₂ + 18 lbs. DDGS

increase efficiency by decreasing input requirements
Commercialization of New Technologies to Improve Fuel-Ethanol Production

1 bushel of corn (56 lbs.)
+ yeast
+ amylase enzymes
+ 92,600 Btu
+ 12 gal water

= 2.7 gallons (18 lbs.) ethanol
+ 18 lbs. CO₂
+ 18 lbs. DDGS

increase efficiency by increasing yield
Commercialization of New Technologies to Improve Fuel-Ethanol Production

1 bushel of corn (56 lbs.)
+ yeast
+ amylase enzymes
+ 92,600 Btu
+ 12 gal water

= 2.7 gallons (18 lbs.) ethanol
+ 18 lbs. CO₂
+ 18 lbs. DDGS

increase coproduct value by improving quality and diversifying products
Front-End Fractionation

fractionation benefits:
• reduce energy requirements
• increase coproduct quality
• increase product diversity
Front-End Fractionation

whole-kernel corn

germ 12% of kernel mass

endosperm 83% of kernel mass (>90% of starch)

bran 5% of kernel mass
Front-End Fractionation

- **whole-kernel corn**
  - germ
    - corn oil
    - high-protein feed
  - endosperm
    - ethanol
    - high-protein DDGS (30-50% lower yield)
  - bran
    - feed
    - fuel (~13,000 to 19,000 BTU/bu)
    - ethanol (starch + cellulose) (increase ethanol yield by ~2 to 3%)
Effects of Front-End Fractionation on DDGS Chemical Characteristics

<table>
<thead>
<tr>
<th>characteristic</th>
<th>concentration (%, w/w dry basis)</th>
<th>conventional</th>
<th>fractionation</th>
</tr>
</thead>
<tbody>
<tr>
<td>moisture</td>
<td>11%</td>
<td>5 to 11%</td>
<td></td>
</tr>
<tr>
<td>crude protein</td>
<td>30%</td>
<td>35 to 45%</td>
<td></td>
</tr>
<tr>
<td>crude fat</td>
<td>11%</td>
<td>3 to 6%</td>
<td></td>
</tr>
<tr>
<td>crude fiber</td>
<td>7%</td>
<td>4 to 8%</td>
<td></td>
</tr>
<tr>
<td>NDF</td>
<td>28 to 38%</td>
<td>24%</td>
<td></td>
</tr>
<tr>
<td>ADF</td>
<td>15%</td>
<td>6%</td>
<td></td>
</tr>
</tbody>
</table>
Motivation for DDGS Research at NCERC

- DDGS is a major coproduct of fuel ethanol produced by fermentation of corn and other starch-based feedstocks in dry-grind plants.
- Main use of DDGS is for cattle feed but has potential for increased use as swine and poultry feed.
- Value of DDGS is determined by its physical and chemical properties:
  - Flowability (transportability)
  - Stability (storage)
  - Nutrient content
  - Palatability
DDGS-Quality Research at NCERC

- survey of DDGS quality characteristics over time in fuel-ethanol industry (DDGS Library)
  - evaluate trends over time within plants and between plants to identify relative importance of factors that control quality

- pilot-scale experiments to identify and evaluate operational factors that affect DDGS quality
  - rigorously evaluate factors believed to affect DDGS quality using an experimental design that allows statistical evaluation of effects
DDGS Library:
Distribution of Crude Protein Concentrations

avg = 30.2 ± 1.8%
(w/w, dry basis)
DDGS Library:
Distribution of Crude Protein Concentrations

avg = 30.2 ± 1.8%
(w/w, dry basis)

DDGS from
front-end
fractionation
DDGS Library: Distribution of Crude Protein Coefficients of Variance

Within-plant CV (%)

Between-plant CV = 5.9%

Fraction of plants

Within-plant CV (%)
DDGS Library:
Distribution of Crude Fat Concentrations

avg = 11.1 ± 1.3%
(w/w, dry basis)

DDGS from front-end fractionation:
avg = 3 to 6%
(w/w, dry basis)

avg = 11.1 ± 1.3%
(w/w, dry basis)
DDGS Library: Distribution of Crude Fat Coefficients of Variance

within-plant CV (%)  fraction of plants

between-plant CV = 11.8%
DDGS Library: Conclusions

• The variation in DDGS chemical composition is larger when different plants are compared than when samples from the same plant are compared over time.

  ➢ This suggests that differences in operation conditions among plants is the greatest source of variation in DDGS composition.

  ➢ Therefore, it might be possible to reduce the variation by identifying and controlling those conditions that most affect specific characteristics.
Experimental Evaluation of DDGS Characteristics: Front End Conditions

corn feed → hammer mill → slurry tank → jet cooker → liquefaction tank → fermentor → to beer well

process water → enzyme → jet cooker

enzyme
Experimental Evaluation of DDGS Characteristics: Front End Conditions

corn feed

hammer mill

screen size

process water

enzyme

jet cooker

liquefaction tank

fermentor

to beer well
Experimental Evaluation of DDGS Characteristics: Front End Conditions

corn feed → hammer mill → slurry tank → jet cooker → liquefaction tank → fermentor → to beer well

- process water
- enzyme
- flow rate
Experimental Evaluation of DDGS Characteristics: Front End Conditions

- Corn feed
- Hammer mill
- Process water
- Enzyme
- Slurry tank
- Jet cooker
- Input rate
- Liquefaction tank
- Fermentor
- To beer well

Temperature
Residence time
Experimental Evaluation of DDGS Characteristics: Front End Conditions

corn feed -> hammer mill -> slurry tank -> jet cooker

process water -> enzyme

enzyme -> liquefaction tank

fermentor -> to beer well
Experimental Evaluation of DDGS Characteristics: Front End Conditions

- Corn feed
- Hammer mill
- Process water
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- Slurry tank
- Temperature
- Residence time
- Jet cooker
- Enzyme
- Input rate
- Liquefaction tank
- Fermentor
- To beer well
Experimental Evaluation of DDGS Characteristics: Back End Conditions

- From distillation
- Whole stillage tank
- Centrifuge
- Thin stillage
- Evaporator
- Syrup
- Wet cake
- Drum dryer
- DDGS
- Recycle DDGS

Flow rate
Experimental Evaluation of DDGS Characteristics: Back End Conditions

- from distillation
- whole stillage tank
- centrifuge
- thin stillage
- evaporator
- syrup
- temperature
- drum dryer
- wet cake
- recycle DDGS
- DDGS
Experimental Evaluation of DDGS Characteristics: Back End Conditions

1. From distillation:
   - Whole stillage tank

2. Centrifuge:
   - Wet cake

3. Thin stillage:
   - Evaporator
   - Syrup addition rate

4. Drum dryer:
   - DDGS

5. Recycle DDGS
Experimental Design for Pilot-Scale Study

• factorial experimental design
  - efficient screening of large number of factors
  - estimate magnitude (and statistical significance) of effects
  - identify interactions among factors
  - provides basis for model development

• fractional factorial design used to test 9 front-end treatment factors at 2 levels

• full factorial design to test 2 back-end factors at 2 levels
  - syrup addition rate covaried with other factors to maintain constant moisture content in dryer input
Effect of Treatment Factors on Protein and Fat Concentrations in DDGS
Prediction of Protein Concentration in DDGS: Multiple Linear Regression

$r^2 = 0.095$
Prediction of Fat Concentration in DDGS: Multiple Linear Regression

\[ r^2 = 0.602 \]
Experimental Evaluation of DDGS Composition: Conclusions

• Some characteristics are strongly affected by operation conditions. So, control is possible (in principle).
  - Post-fermentation conditions (e.g., drying and evaporation) appear to be most important.

• Other factors, which have not yet been identified, are also important.
  - Effects are highly interactive and (possibly) nonlinear.
Effects of Microbial Contamination on Ethanol Yield

• Acetic-acid bacteria aerobically oxidize ethanol to acetic acid
  ➢ acetic acid inhibits yeast growth by 80% at concentration of 0.8%
  ➢ AAB grow mainly in yeast propagation tanks and beer well

• Lactic-acid bacteria anaerobically ferment glucose to lactic acid or lactic acid plus ethanol (or acetic acid)
  ➢ lactic acid inhibits yeast growth by 80% at concentration of 3.8%
  ➢ LAB compete for glucose and other nutrients in fermentors

• Reduction in ethanol yield is proportional to extent of bacterial contamination
Control of Microbial Contamination

• Cooking
  ➢ heating to >80 °C (>176 °F) for >10 minutes will kill most vegetative cells

• Cleaning and sanitizing
  ➢ hot caustic CIP (>55 °C) effectively kills most vegetative cells

• Antibiotics
  ➢ selective for prokaryotes (bacteria) over eukaryotes (yeast)
  ➢ penicillin G and virginiamycin are most common
  ➢ concern over antibiotic residues in DDGS

• Other antimicrobial compounds
  ➢ bacteria must be more sensitive than yeast
  ➢ examples include oxidizers (e.g., chlorine dioxide) and natural products (e.g., α and β acids from hops)
Hop-Derived Antimicrobial Products

• Hops have been used for centuries to control bacterial growth in beer (and impart desirable flavor and aroma)
  ➢ most effective against Gram-positive bacteria (e.g., LAB)

• Antimicrobial properties of hops are due to alpha and beta acids contained in the resins
  ➢ alpha and beta acids can be extracted from lupulin glands using supercritical carbon dioxide
  ➢ alpha acids are isomerized by heating; iso-α-acids are more effective antibacterial agents
Effect of Lactic-Acid Bacteria on Ethanol Production Kinetics

- Yeast only
- Yeast + L. plantarum

Time (hrs): 0 1 2 3 4 5
Ethanol concentration (g/100 ml): 0 2 4 6 8 10

Graph shows the comparison of ethanol production kinetics between yeast only and yeast plus L. plantarum.
Ethanol Production Kinetics

- Yeast only
- Yeast + *L. plantarum*
- Yeast + LAB + Antibiotic

Ethanol concentration (g/100 ml) vs. time (hrs)
Ethanol Production Kinetics

- Yeast only
- *L. plantarum*
- Antibiotic
- Hop A (50 ppm)

**Axes:**
- **Y-axis:** Ethanol concentration (g/100 ml)
- **X-axis:** Time (hrs)
Lactic Acid Production Kinetics

Yeast only vs. yeast + L. plantarum

Time (hrs) vs. Lactic Acid Concentration (g/100 ml)

- Yeast only
- Yeast + L. plantarum
Lactic Acid Production Kinetics

![Graph showing lactic acid production kinetics with time and concentration](image-url)
Lactic Acid Production Kinetics

- Yeast only
- *L. plantarum*
- Antibiotic
- Hop A (50 ppm)

Lactic acid concentration (g/100 ml) vs. time (hrs)
Alternatives to Antibiotics: Conclusions

• Concern over the presence of antibiotic residues in DDGS is growing. Alternatives to antibiotics are available, for example:
  ➢ cooking and cleaning are important preventative procedures
  ➢ chemical oxidants and natural products from other applications have been adapted for use in fuel-ethanol production
    ⇒ bacteria must be more sensitive than yeast

• Some hop-derived products can be more effective than common antibiotics
  ➢ not all hop products are equally effective
    ⇒ alpha acids are more effective than beta acids
    ⇒ reduced alpha acids are most effective

• Other alternatives are under development
Role of NCERC in Technology Development

• NCERC was created to provide a facility in which technology developers from industry, academia, and government can evaluate new products and equipment under realistic conditions.
  ➢ NCERC is open to all on a fee-for-service basis
  ➢ NCERC has strict confidentiality policies to protect proprietary information
  ➢ studies are designed to ensure that clients retain their intellectual property rights

• NCERC provides independent and unbiased evaluations of the performance of new technologies.

• NCERC is supported by client fees along with state and federal funding.
  ➢ state and federal funding is determined year-to-year