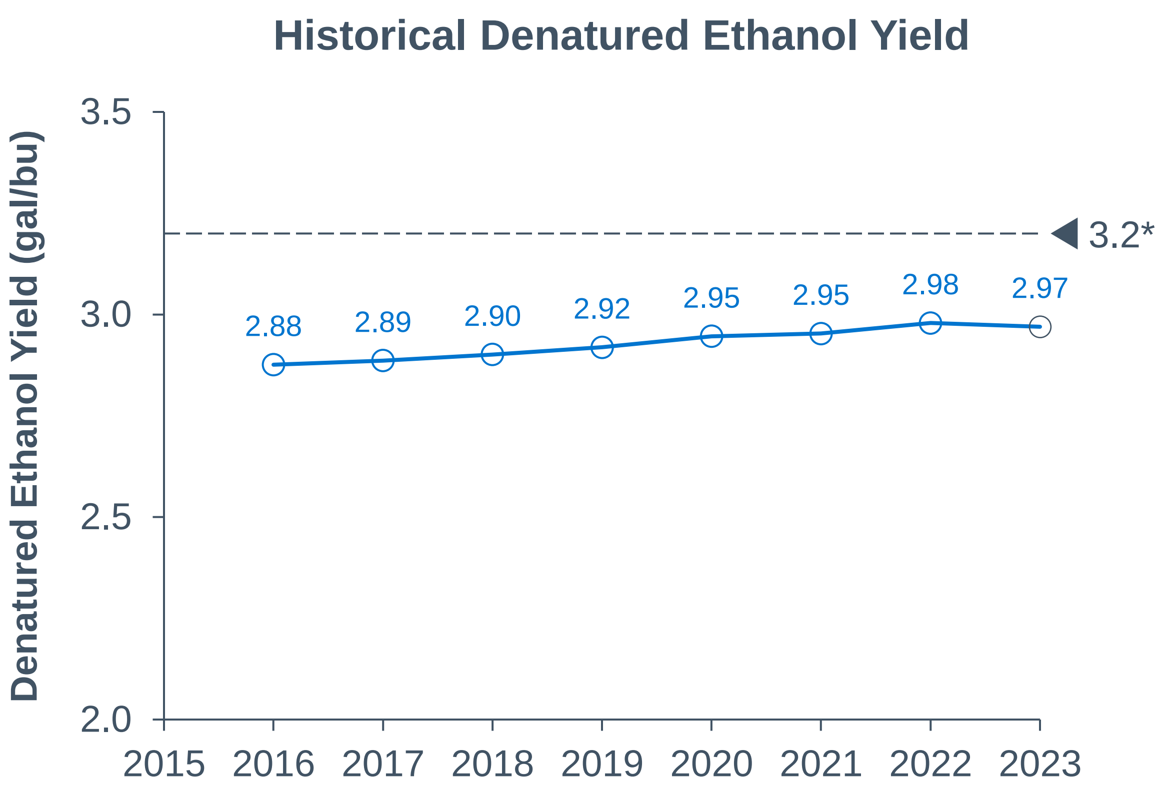
XCELIS® Accessories

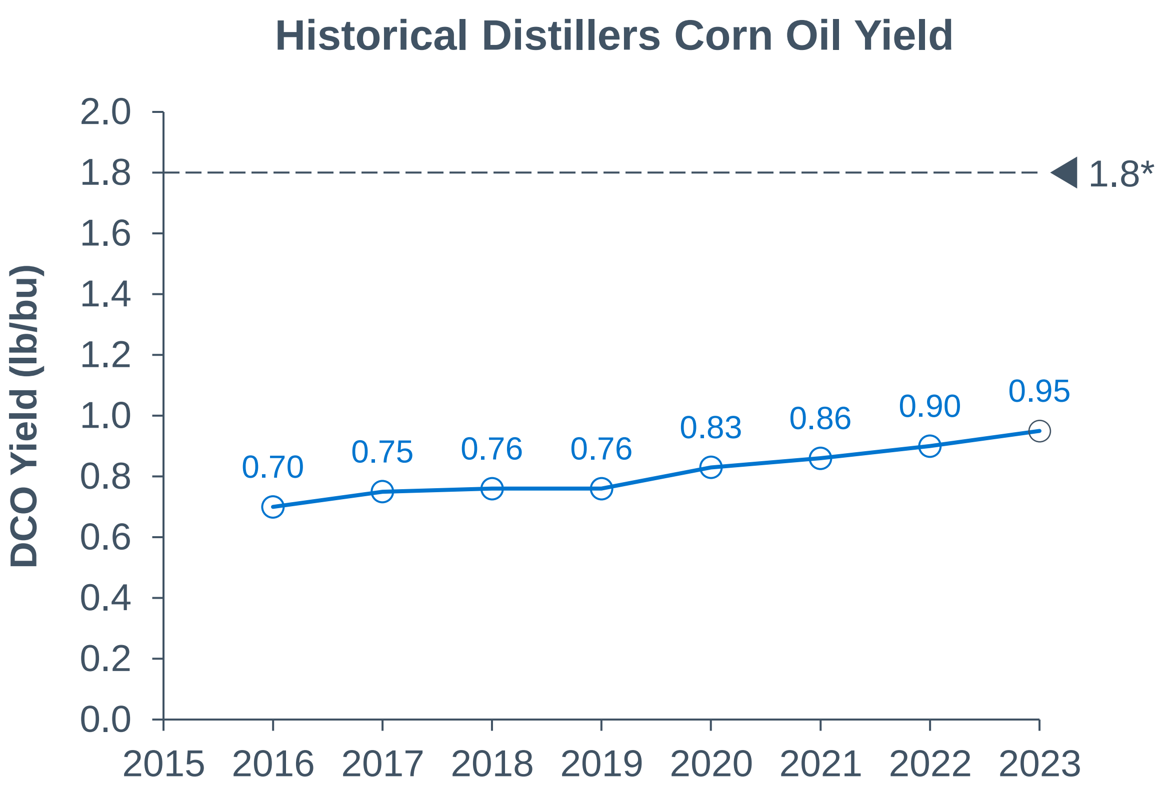
MAXIMIZING OIL YIELD: CUTTING-EDGE TECHNIQUES IN MEASUREMENT, MONITORING, & ENZYME-DRIVEN YIELD OPTIMIZATION

Mary Beth Willems – North America Application Lead

TRENDS IN DISTILLERS CORN OIL YIELDS

Still significant room for growth in Distiller's Corn Oil (DCO) yield





***Theoretical maximum yield**

https://usda.library.cornell.edu/concern/publications/n583xt96p?locale=en#release-items

TODAY'S TALK

1. **Fundamentals of corn oil and its recovery**
   a) Oil's native biochemical structure in plants: Oleosomes
   b) The different forms of oil
   c) The role that solids play
2. **Oil measurement**
   a) Oil mapping & estimating centrifuge oil efficiency
   b) Oil measurement methods
3. **The use of cellulases and xylanases to increase oil production**
   a) Trial setup
   b) Monitoring downstream performance
   c) Oil yield performance
4. **Summary**

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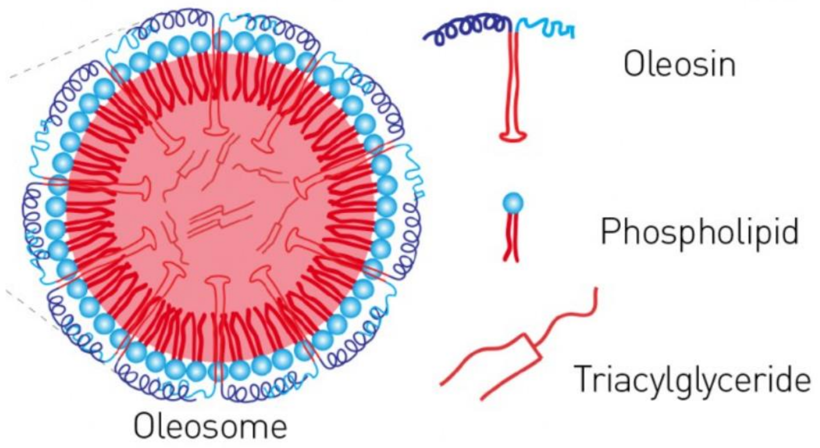
FUNDAMENTALS OF OIL

NATIVE STRUCTURE OF OIL IN CORN PLANT

- Oil is packaged in tight little spherical droplets called **oleosomes**
- In native oleosome structure, the triacylglyerides are protected by protein (oleosin) and phospholipids



Zielbauer et al.: *From fat droplets in plant cells to novel foods*. http://www.int.laborundmore.com/ archive/936206/From-fat-droplets-in-plant-cells-to-novel-foods.html
Nikiforidis and Kiosseoglou. 2009. *Aqueous Extraction of Oil Bodies from Maize Germ (Zea mays) and Characterization of the Resulting Natural Oil-in-Water Emulsion*

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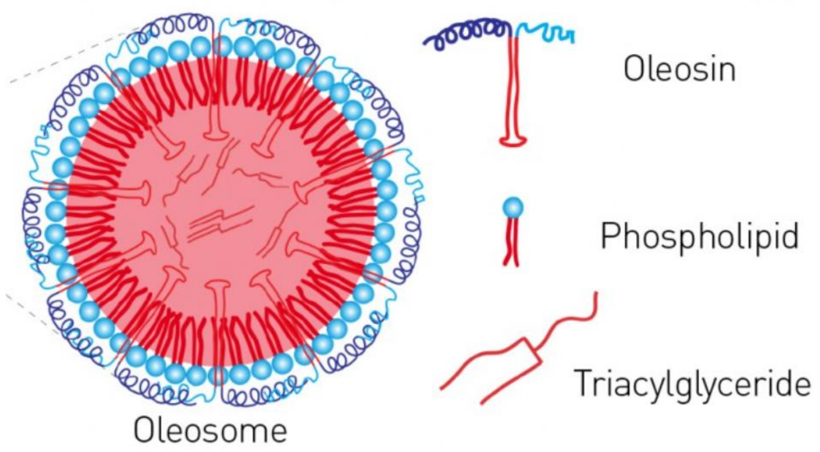
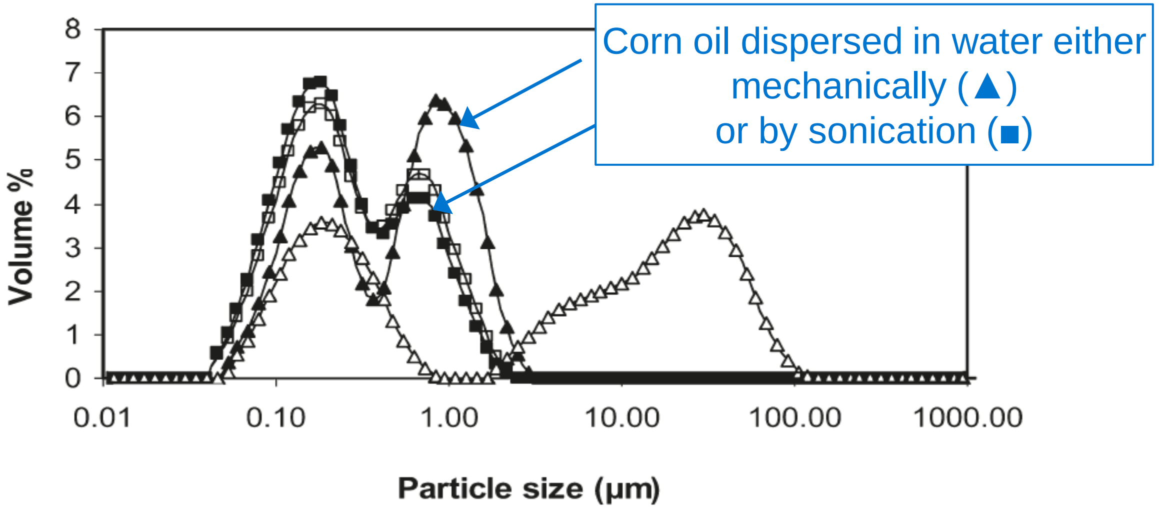


- Oleosomes are small droplets, with an average diameter of < 2 µm
- At this native size, oil cannot be recovered by a tricanter or disc stack. You must disturb the native structure to make the oil recoverable
- Oleosomes are embedded within the cellulose/hemicellulose/protein cell wall matrix



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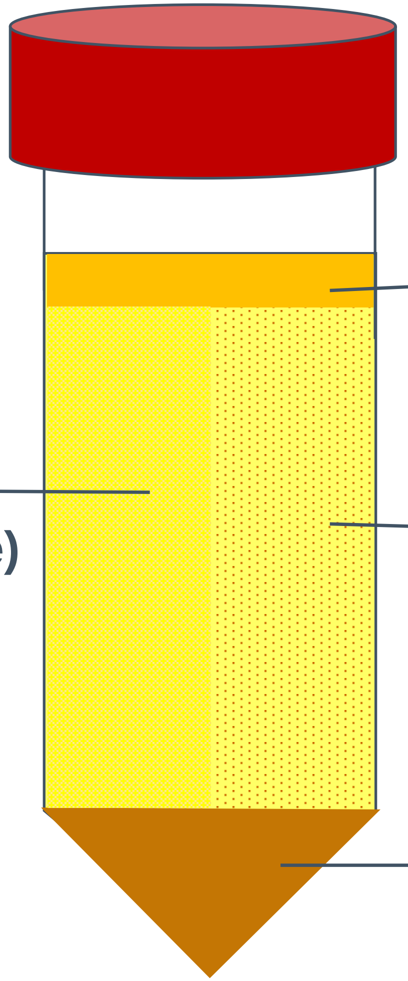
DIFFERENT FORMS OF OIL



THE ROLE OF WET CAKE SOLIDS IN CORN OIL RECOVERY

- **The majority of unrecovered distillers corn oil is lost to the wet cake**
- **Wet cake solids act like a sponge, soaking up free oil, and making it unrecoverable**

Added wet cake solids:

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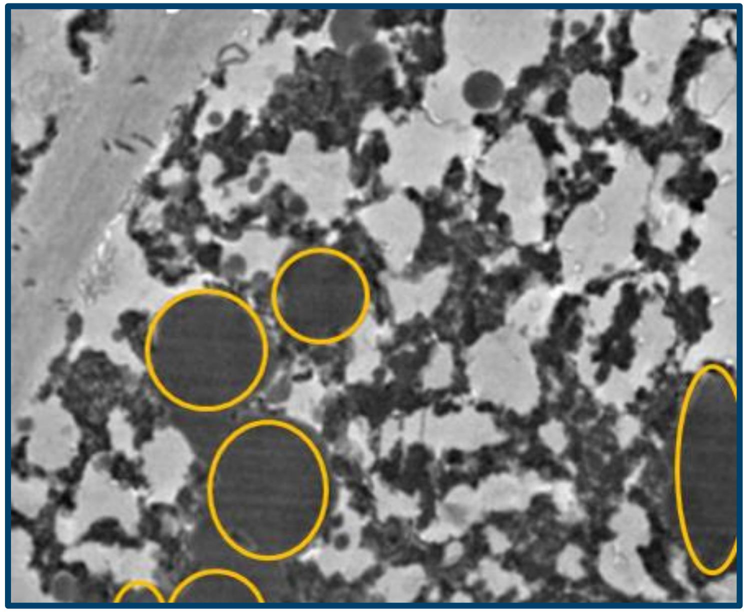
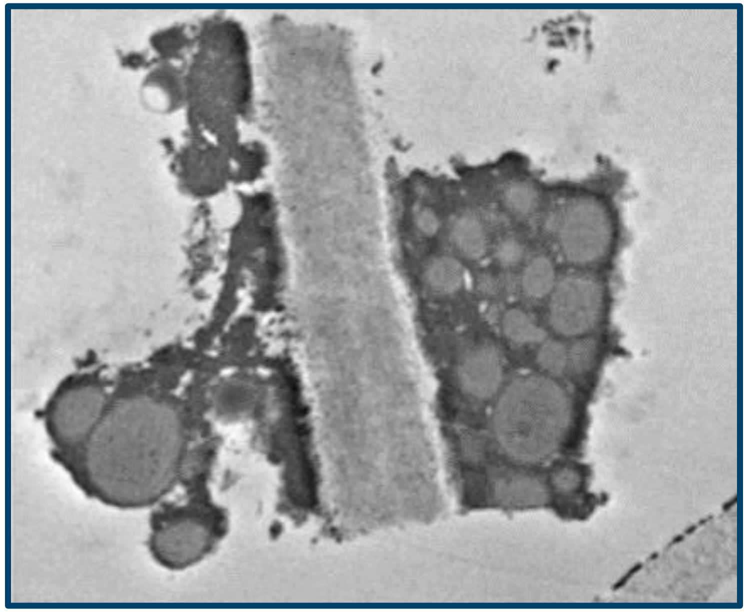
THE ROLE OF SOLIDS IN CORN OIL RECOVERY

Syrup



Syrup after mechanical disruption



Syrup after enzyme treatment

- Oil droplets stick to fiber, as well as intact and denatured protein
- This likely limits the recovery of DCO by tricanter or disc stack

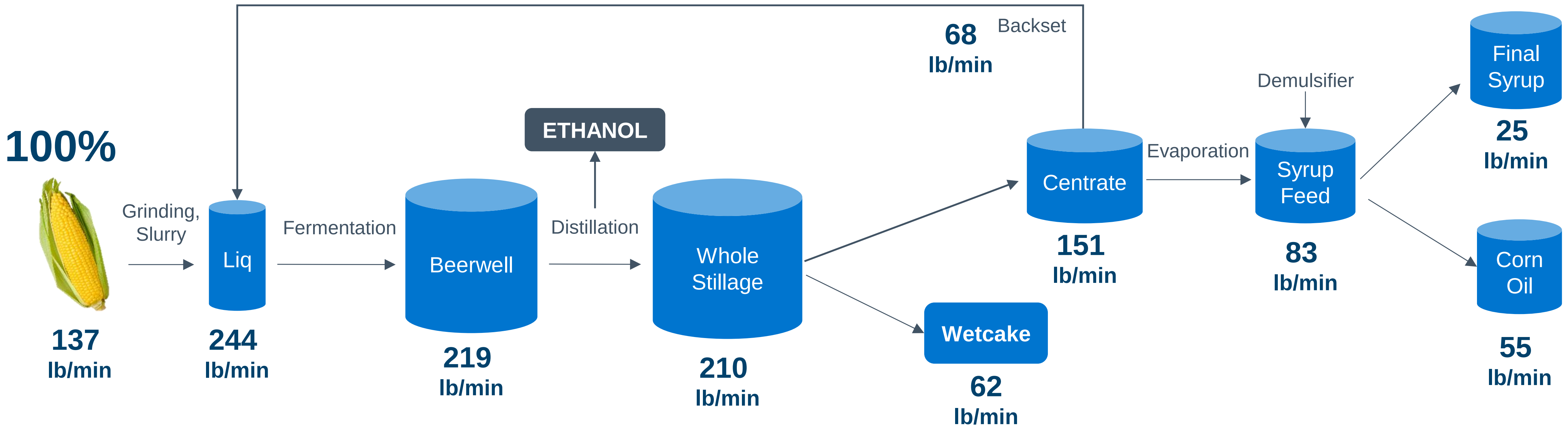
- As fiber- and protein-rich solids are enzymatically broken down, small oil droplets can aggregate to form larger ones. These larger oil droplets are more easily recovered by a corn oil centrifuge

Majoni et al. 2011. *Enzyme Treatments to Enhance Oil Recovery from Condensed Corn Distillers Solubles*.

OIL MEASUREMENT

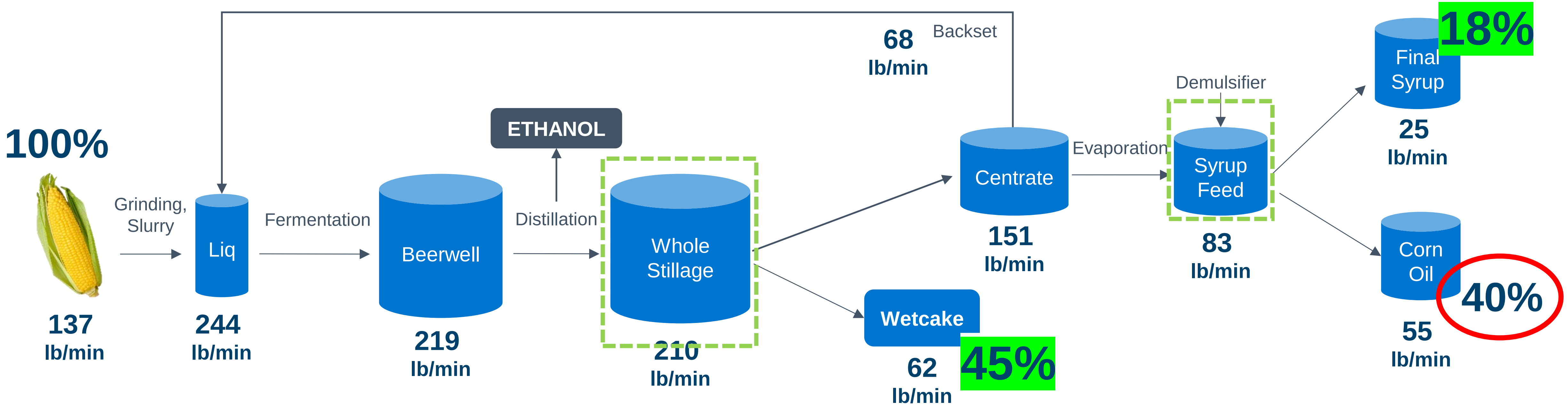
WHAT IS A COMPLETE OIL MAPPING?

- An oil mass balance
- A complete oil mapping details the flow of oil throughout the entire plant to see where it is moving:



WHY IS THIS VALUABLE?

- Oil mapping can be used as a tool to identify how much oil is lost and where, including to backset
  - Oil going with wet cake and de-oiled syrup (or heavy phase) cannot be recovered
- Oil mapping can also be used to estimate a plant's oil yield at a specific point in time
- In the example below, 45% of oil is lost to wet cake and 18% is lost to de-oiled syrup (heavy phase), respectively. Additionally, we can estimate that this plant was achieving ~40% oil yield.



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CENTRIFUGE EFFICIENCIES 101

- In dry grind process, oil is mainly lost at two places: decanter centrifuge and COSS
- We introduced a parameter named centrifuge efficiency to determine the amount of oil recovered in a centrifuge
- In other words, efficiencies would tell us how much oil is **not** lost to wet cake or de-oiled syrup

- Centrifuge efficiency is the ratio of oil flow in product of interest to oil flow to centrifuge inlet
- Decanter centrifuge efficiency is defined as ratio of oil flow in centrate to oil flow in whole stillage
- COSS efficiency is defined as ratio of oil flow in oil product stream to oil flow in syrup

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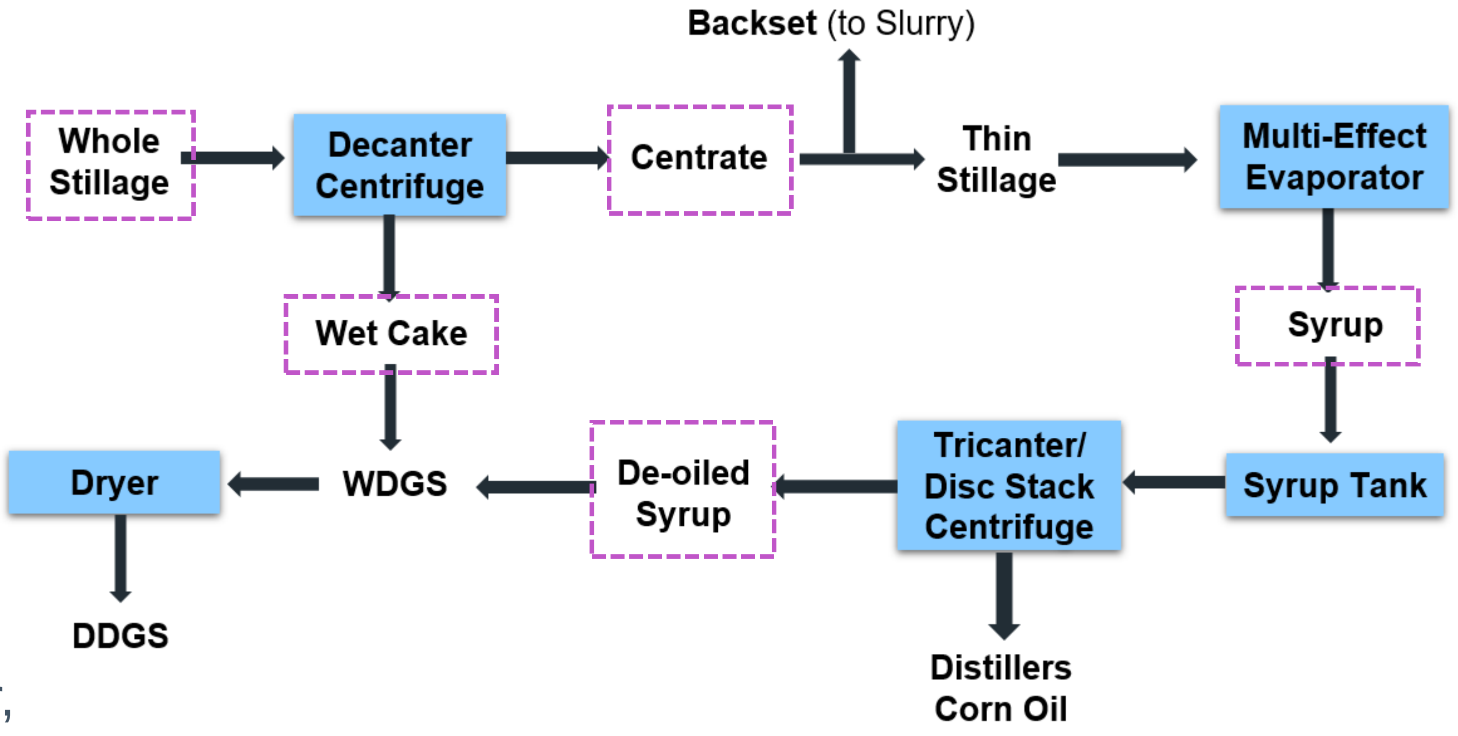
ESTIMATING CENTRIFUGE OIL EFFICIENCY

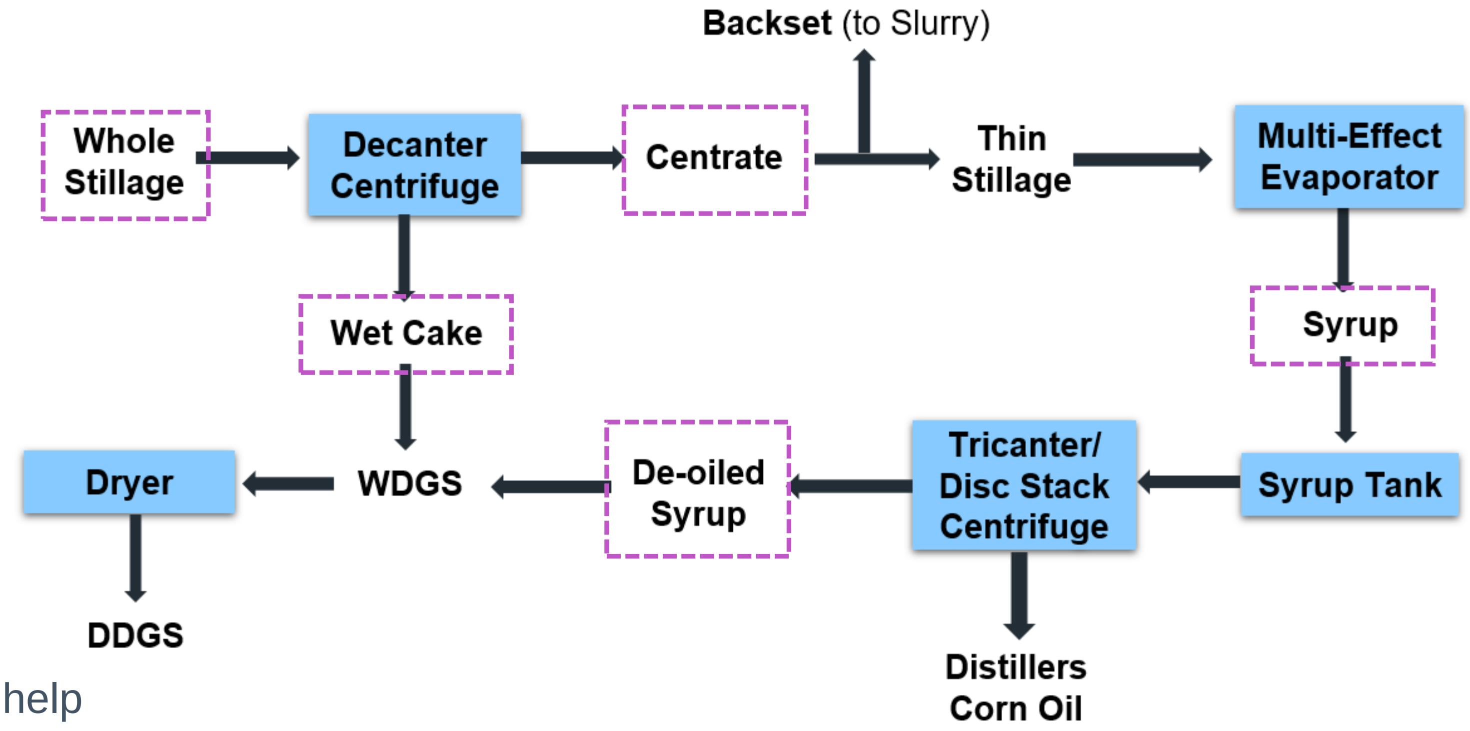
- **What needs to be measured to calculate efficiencies?**
  - Solids
  - Oil contents
  - Flow rates

- **Perform oil mappings before and after changes to measure effectiveness of the change:**
  - Enzyme addition or dose change
  - Centrifuge adjustments (decanter, tricanter, disc stack)



ESTIMATING CENTRIFUGE OIL EFFICIENCY

- The efficiency with which decanter and tricanter/disc stack separates oil can be estimated by determining fat content and dry solids on downstream samples:
  - Whole stillage
  - Centrate
  - Wet cake (without syrup)
  - Syrup feed to corn oil centrifuge
  - Heavy Phase (off each machine)
- Process information:
  - Flowrates of each stream, wherever possible



- Fat measurements of downstream samples can help identify the impact of process or enzyme changes, and potentially troubleshoot if needed

TRADITIONAL OIL QUANTIFICATION METHODS

- Traditional fat quantification methods involve organic solvent extraction combined with gravimetric analysis
- They are tedious, time-consuming, but have been the benchmark for decades and provide reasonable data
- 3rd party labs usually have 2 fat determination methods:

Fat by Acid Hydrolysis (A)
Method reference: AOAC 954.02 mod.; AOAC 922.06 mod. ; AOAC 925.32; AOAC 933.05, modified
Optimal / Minimal Sample Quantity: 20 g / 10 g
(Local Code: FAT_AH_S;DEFAULT)

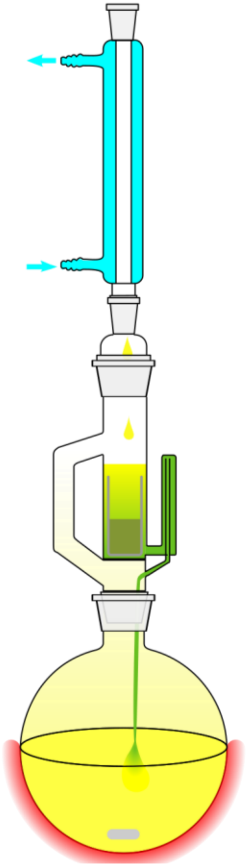
Technical description of test **[FS0AI]**

Name	Fat by Acid Hydrolysis	
Description	The sample is hydrolyzed with hydrochloric acid. The fat is extracted using ether and hexane. The extract is filtered through a sodium sulfate column. The solvent is evaporated from the remaining extract and the fat is dried and weighed.	
ApplicableOn	Food, feed products, meat, cheese, and eggs	
Method	Gravimetry	
Parameter	**Quantification limit limit(LOQ)**	**CAS Number**
[7027A006] Fat	0.1 %/No unit	57-10-3

Fat (Soxhlet method)
Method reference: AOAC 960.39 & 948.22 (modified)
Optimal / Minimal Sample Quantity: 20 g / 10 g
(Local Code: FSOX_S;DEFAULT)

Technical description of test **[FS153]**

Name	Fat (Soxhlet method)	
Description	The sample is weighed into a cellulose thimble containing sand or sodium sulfate. The thimble is dried to remove excess moisture. Pentane is dripped through the sample to remove the fat. The extract is then evaporated, dried, and weighed.	
ApplicableOn	Meats, feed products, seeds, and nuts	
Method	Gravimetry	
Parameter	**Quantification limit limit(LOQ)**	**CAS Number**
[7027A006] Fat	0.1 %/No unit	57-10-3



https://chembam.com/definitions/soxhlet-extractor/
https://www.eurofinsus.com/food-testing/resources/crude-fat-analysis-frequently-asked-questions/#:~:text=Answer%3A%20This%20method%20is%20used,for%20a%20variety%20of%20foods.

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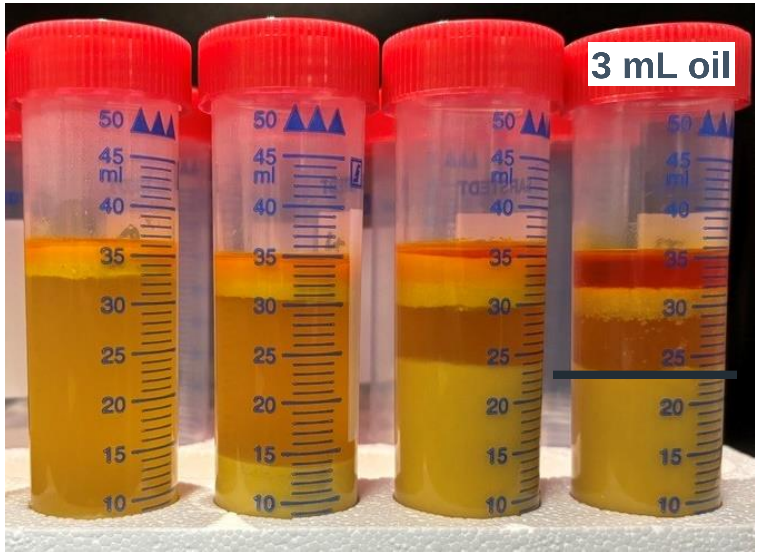
IN-HOUSE OIL MEASUREMENT – SYRUP SPINS

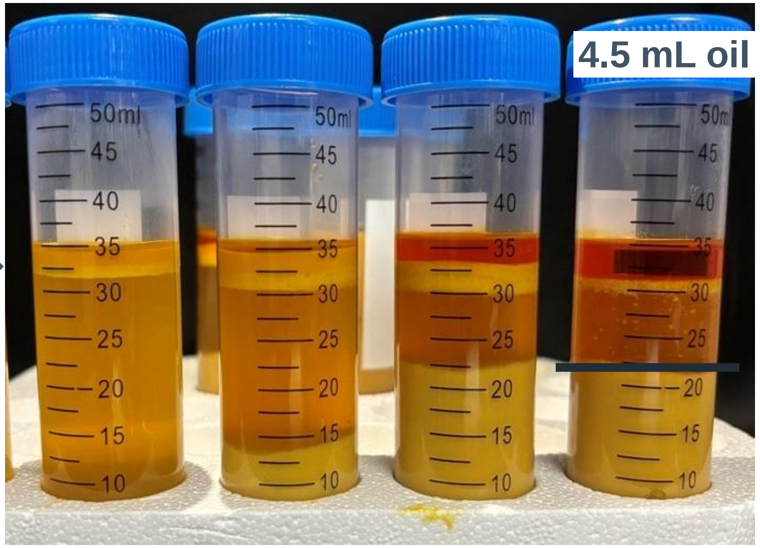
- Crude quantitative method, gives you free oil, but not oil in aqueous or solids phases
- With the introduction of cellulase/xylanase, you can visually observe an increase in volume of free oil in syrup spins
- Additionally, you observe more free oil appearing prior to demulsifier addition, and in earlier evaps

Baseline:
small amount of xylanase



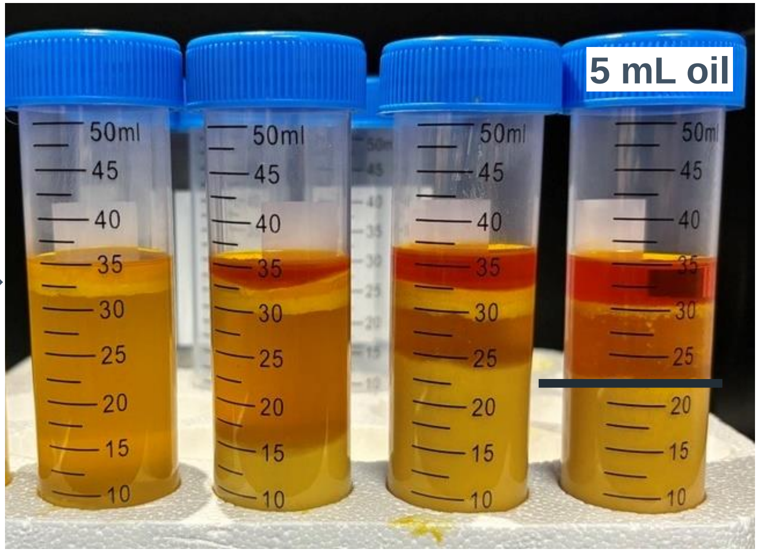
Evap 6 Syrup | Evap 7 Syrup | Evap 8 Syrup | Syrup Feed +demulsifier

Trial phase:
2x xylanase + cellulase
1st round of drops



Evap 6 Syrup | Evap 7 Syrup | Evap 8 Syrup | Syrup Feed +demulsifier

Trial phase:
2x gal xylanase + cellulase
Start of 2nd round of drops



Evap 6 Syrup | Evap 7 Syrup | Evap 8 Syrup | Syrup Feed +demulsifier

Photos property of IFF

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IMPROVING OIL QUANTIFICATION METHODS

- “Low resolution” NMR has the potential to deliver reproducible and very precise oil data on a range of sample types
- This approach measures only fat molecules, and is more specific than techniques involving chemical extraction
- It is also fast, giving results within minutes

Benchtop H-NMR – Total Oil Content

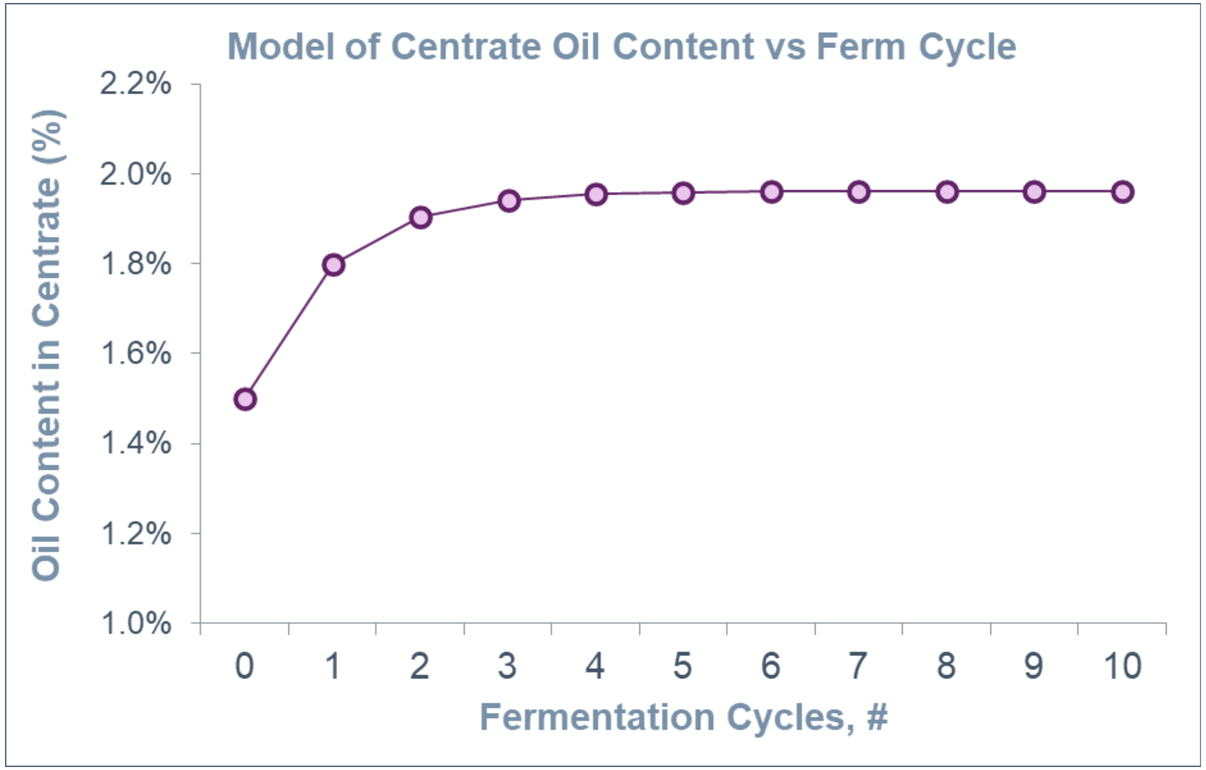
Photo property of IFF

CELLULASES & XYLANASES

TRIAL - SETUP

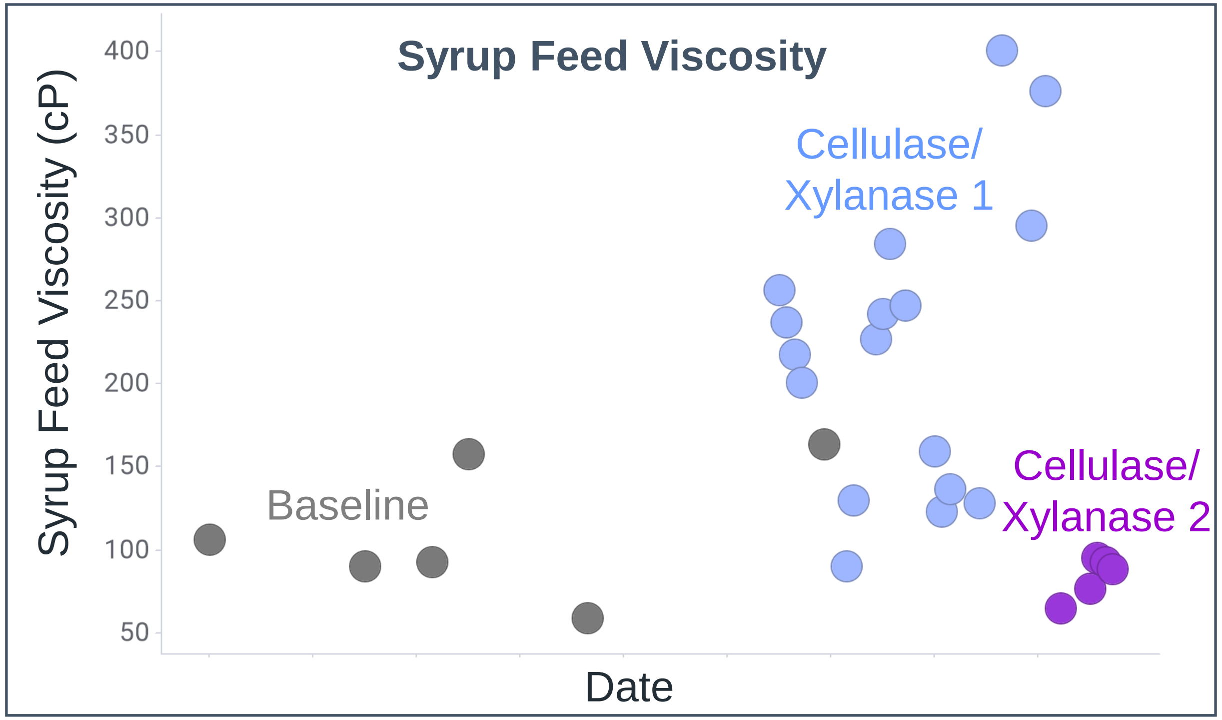
- Multi-week baseline evaluation
  - Centrate solids profile
  - Syrup viscosity
  - Corn oil centrifuge performance
  - Oil yield performance

- 2 trial phases
  – Cellulase/Xylanase 1 Trial
  – Cellulase/Xylanase 2 Trial



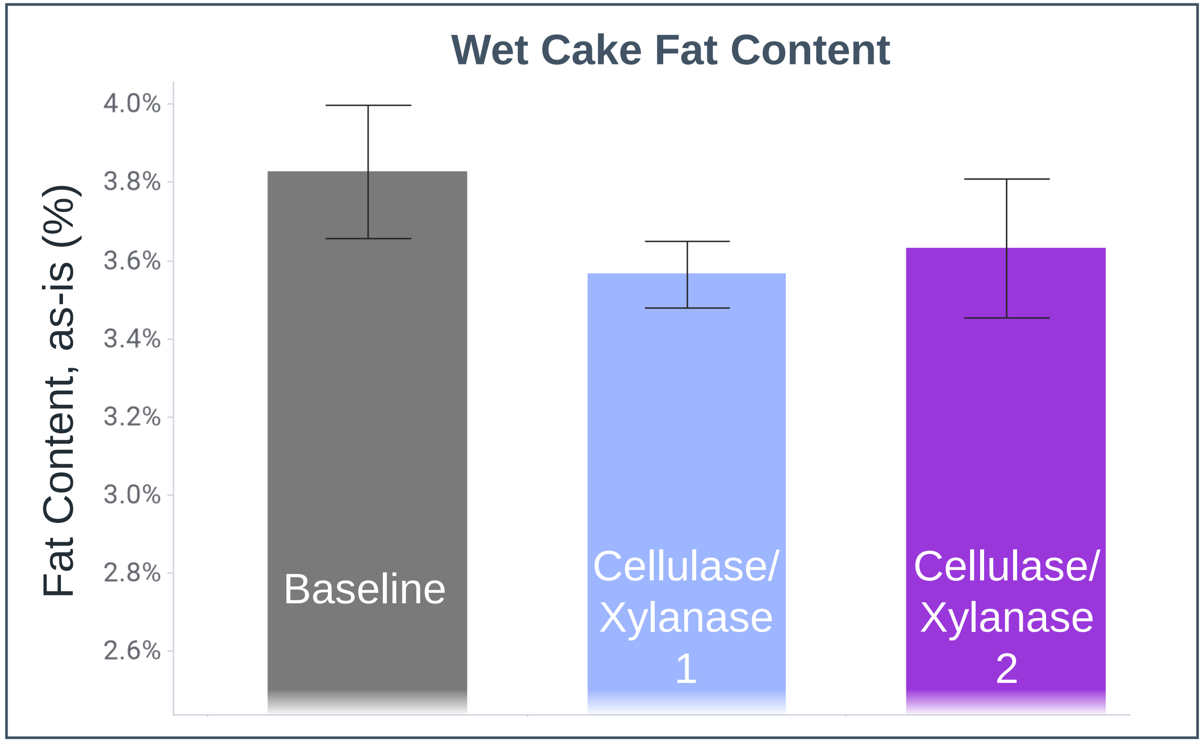
MONITORING VISCOSITY

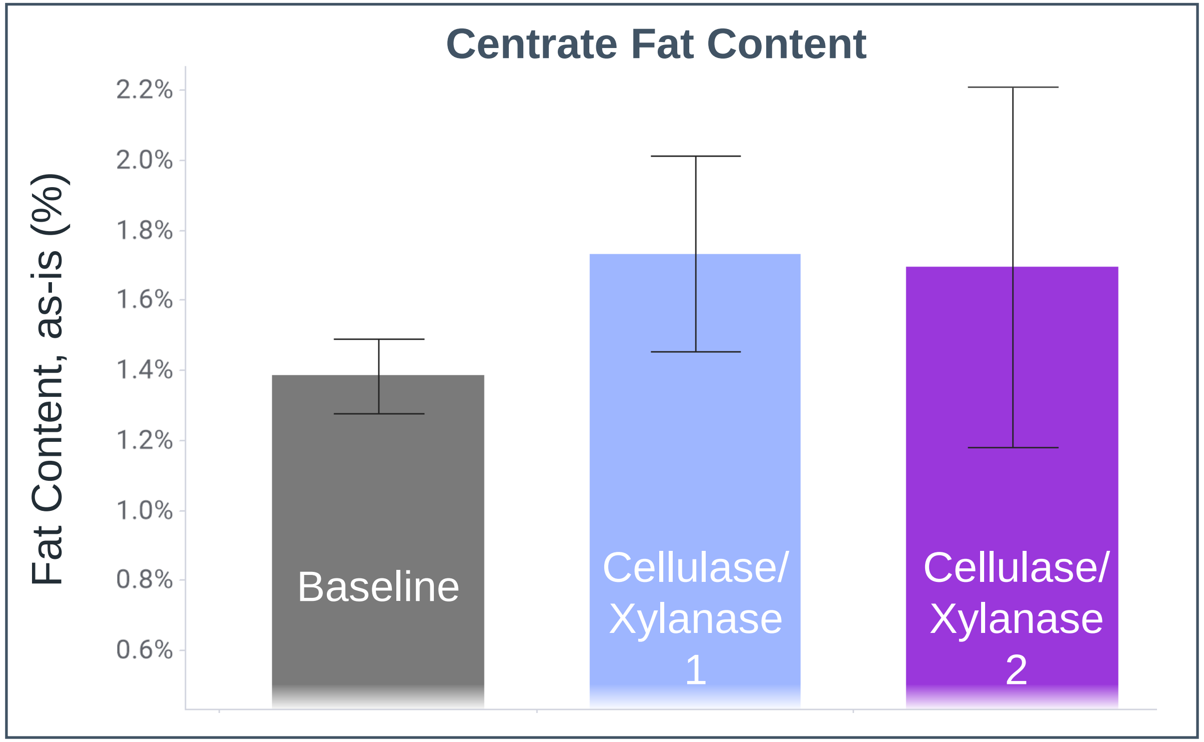
- Establishing baseline syrup viscosity is important in selecting enzyme dosing
- During early phase of trial, daily or weekly monitoring of solids and syrup viscosity can be critical
- Moderate increases to syrup viscosity were observed, but remained well within acceptable range
- No negative process impacts were noted by plant personnel during trial



MONITORING FAT CONTENT DOWNSTREAM

- With cellulases/xylanases, **wet cake fat** (as-is) should maintain/**decrease**; whereas **centrate fat** should maintain/**increase**





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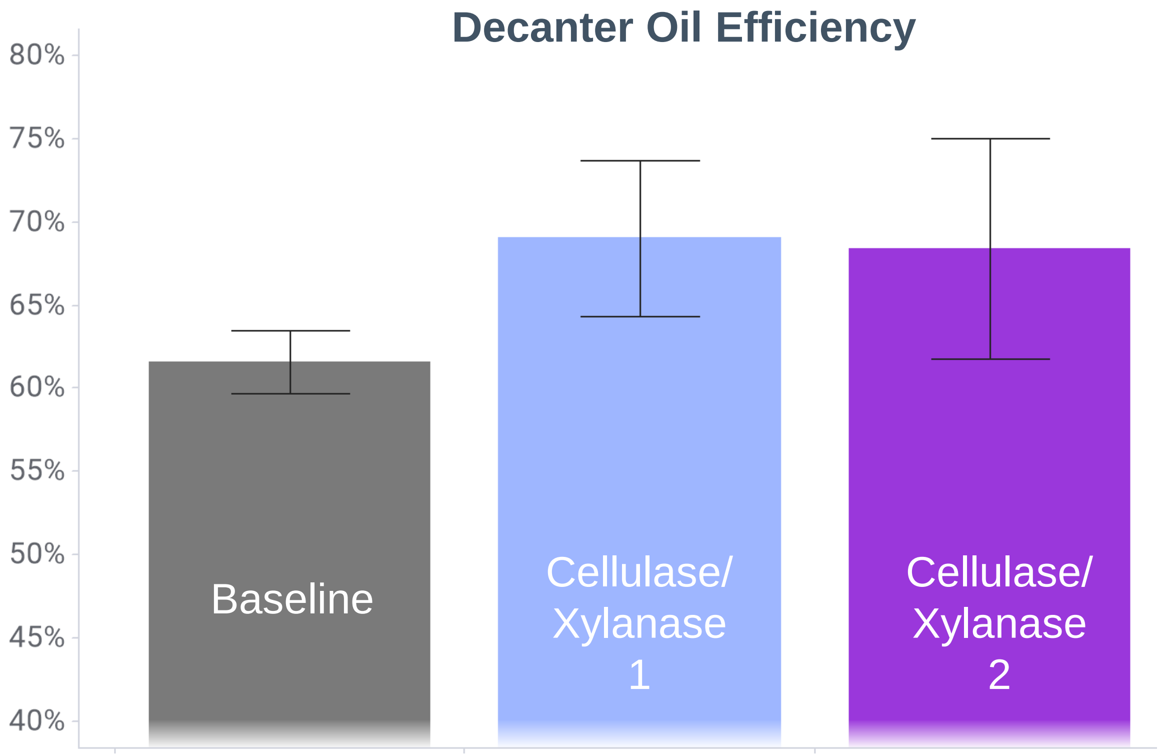
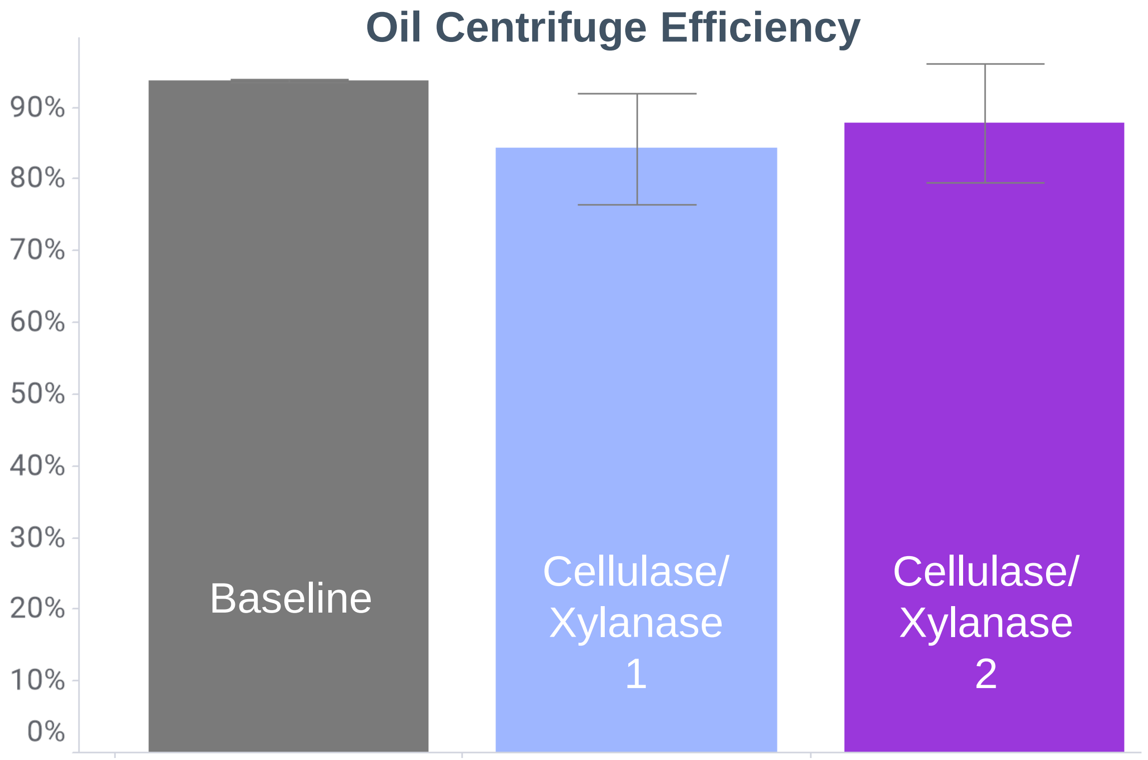
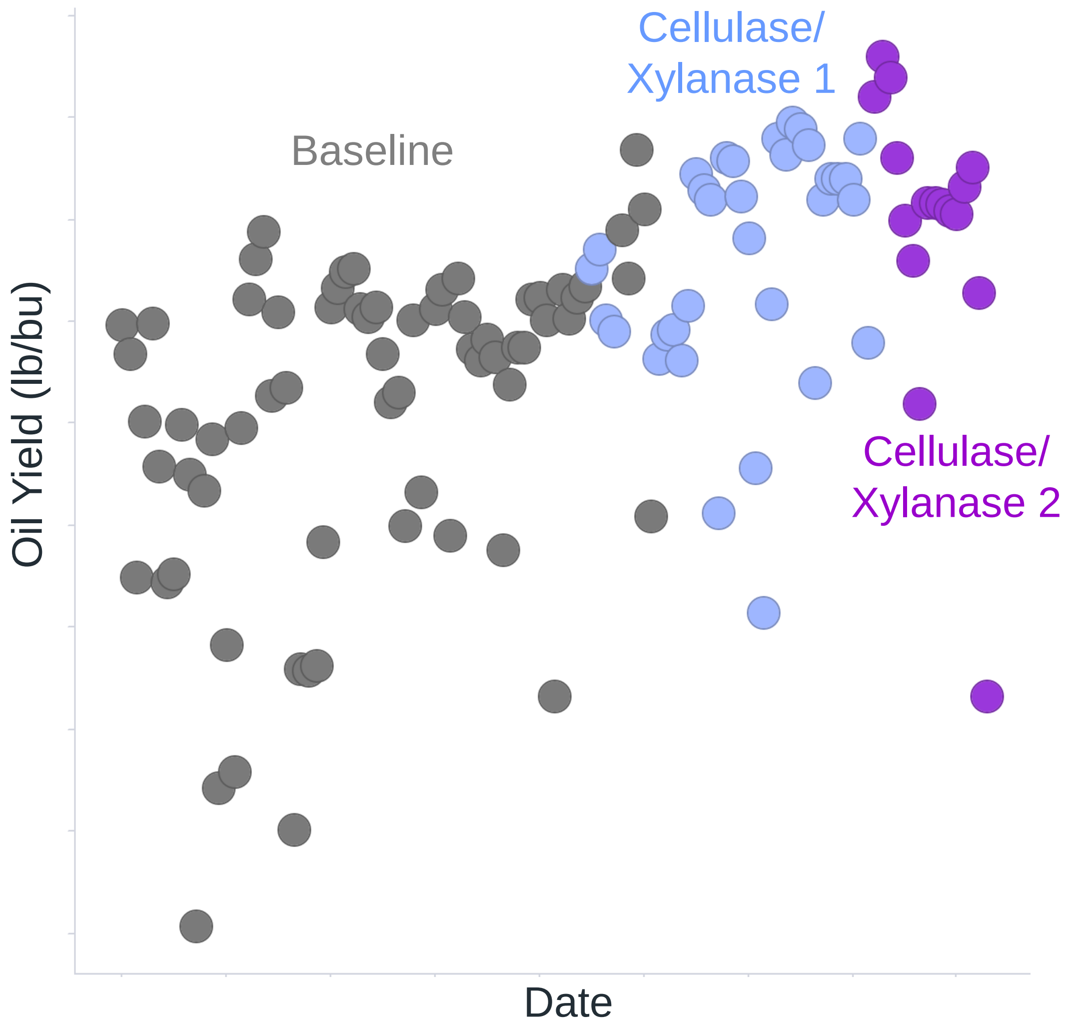
CENTRIFUGE OIL EFFICIENCY

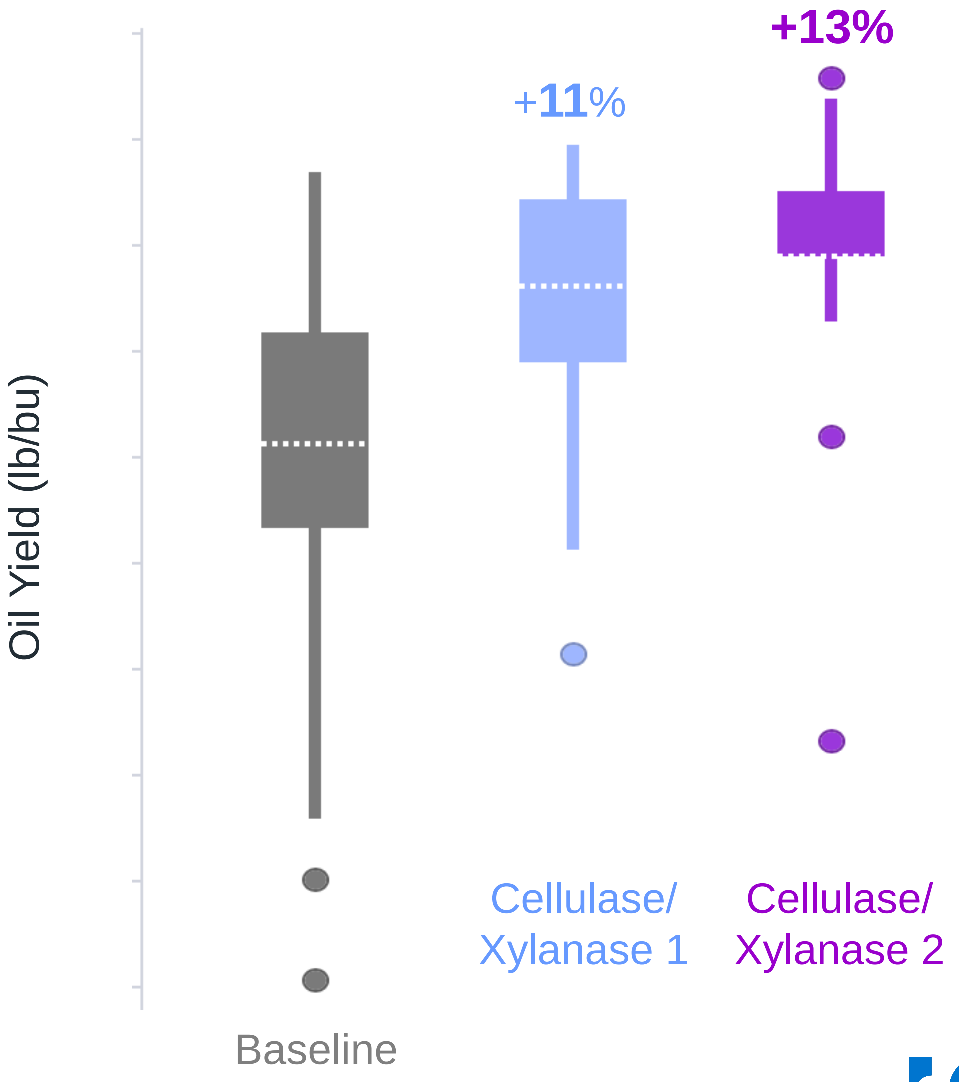
- **The addition of xylanases and/or cellulases increased the decanter oil efficiency**
- The oil centrifuge efficiency dropped slightly during the trial, due to unrelated issues with the machines
- After fixing, oil efficiency started to increase again, suggesting there is still further room for yield improvement





OIL YIELDS IMPROVED 11-13% WITH CELLULASE AND XYLANASE





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SUMMARY

- **Oil measurement methods are getting faster and more accurate**
- **Most oil is lost in the centrifuges (decanters, tricanters, disc stacks)**
- **Oil mapping will calculate the amount of oil lost at each step**
  - Performing multiple mappings can help determine the effectiveness of changes
- **It is important to evaluate and understand baseline performance metrics prior to a cellulase/xylanase trial, as they will dictate recommended enzyme dosing regime. Monitoring and supporting an oil trial is critical. It allows for faster troubleshooting and dosing/process optimization.**
- **Enzyme additions as well as process adjustments can move more oil into a recoverable state**
- **Addition of IFF xylanase and/or cellulase has <u>increased oil yield up to 15%</u>**

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THANK YOU!

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