Rahr Technical Center

Technical Capabilities and Case Studies on Beer QA Challenges

Presented to

Rahr/BSG Malt U

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July 20, 2018

Minneapolis, MN

Agenda:

1. Introduction
2. Technical Capabilities
3. Case Studies
   - Yeast performance factors
   - Beer haze characterization
   - Beer flavor stability investigation
   - Starch gelatinization properties by RVA

Rahr Technical Center

- Quality Control:
  - Malt
  - Barley
  - Hops
  - Other brewing ingredients
- Beer Analytical Lab
- Pilot and Micro-malting Lab
- Materials R &D Analytical Lab
- Sensory Tasting Booths
- Research Brewery

Malt Quality Lab

Barley Quality Lab

Micro-Maltings
Joe-White and Phoenix machines

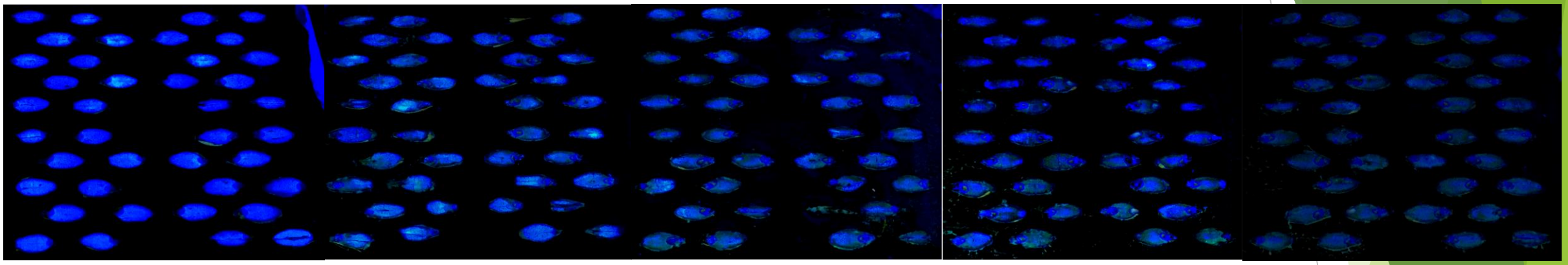
(up to 80 samples/batch)

Pilot-Maltings (under manufacturing)

(up to 2 batches/week @ 140kg malt/batch)

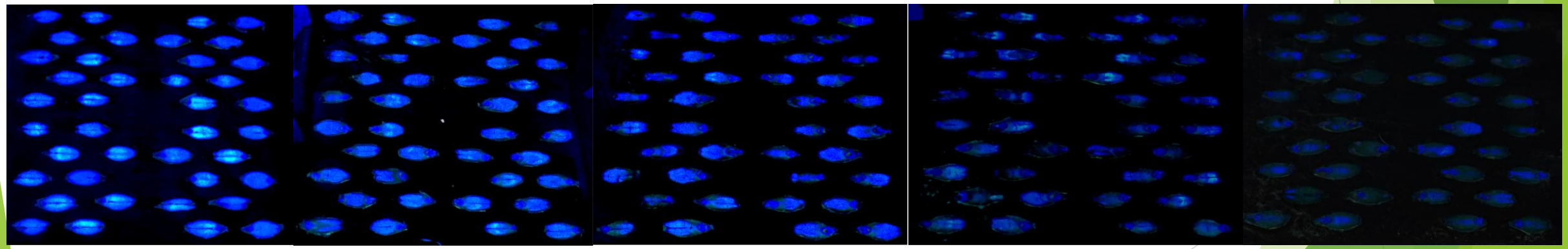
Malt Modification by Microfluo Malt Analyser

Pinnacle



Pinnacle	Barley	Day 1	Day 2	Day 3	Day 4
Modification	76.7	80.7	94.9	98.0	99.7
Homogeneity	59.8	66.4	82.1	90.5	96.9

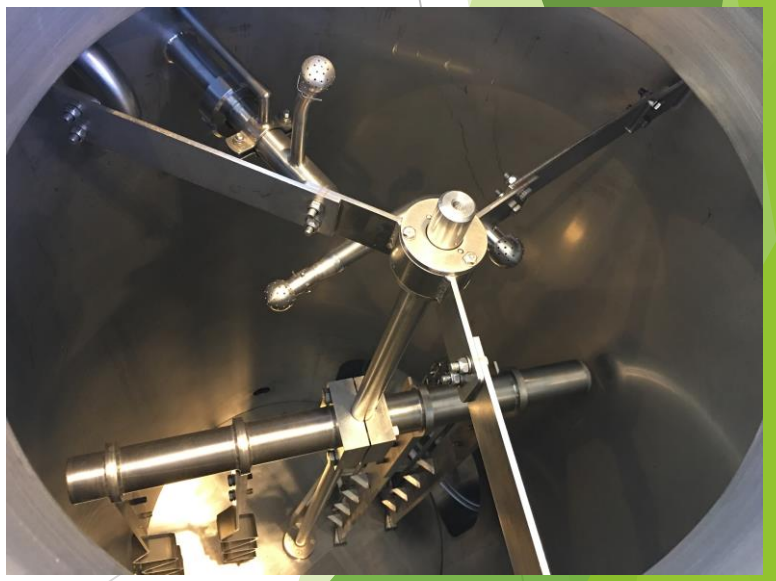
Copeland



Pinnacle	Barley	Day 1	Day 2	Day 3	Day 4
Modification	9.2	56.4	76.1	88.7	97.2
Homogeneity	62.3	63.6	65.4	72.8	86.7

Research Brewery

- Esau Huber 3 HL Brew house
- 48 HL fermentation capacity(+28.5 HL in Aug'18) :
  - 7 x 3 HL (expanding by 6x3HL in Aug 2018)
  - 3 x 6 HL
  - 6 x 1.5HL (expanding by 7x1.5 HL in Aug 2018)



Beer Analytical Lab

- Anton Paar Alcolyzer Beer ME
  - Determines alcohol, density, original extract, real extract, degree of fermentation, calories
- Pentair Nibem Foam Analyzer
- Pentair C-TPO packaged oxygen analyzer
- Pentair Vos Rota Turbidity meter
- Spectroquant: polyphenols, BU's, iron and other basic beer parameters

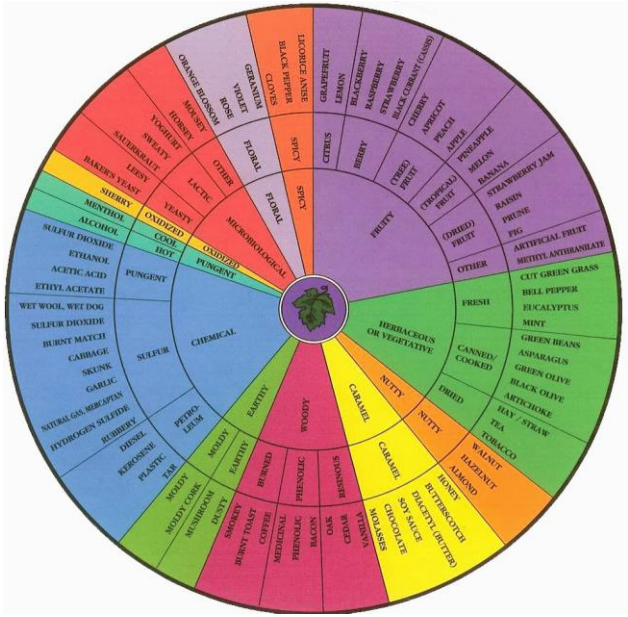
Data management: Sample Manager in conjunction with QC results

R&D Analytical Lab

- GC-MS-TOF for Flavor Analysis
  - SPME
  - TWISTER/TDU
  - NDMA
- GC-FPD: DMS/DMSP
- GC-ECD: DON and VDK
- HPLC
  - DAD – Hop Compounds and other non polar/polar
  - RID – Carbohydrates and Size Exclusion
  - FLD – Amino Acids and others
- PYF – Laminar Flow hood
- Yeast Cellometer – yeast count, viability, vitality
- Bench (10 L) and bottle scale fermentation
- Hop Oil Distillation (QC lab) and hop oil analysis
- RVA: (Starch) Pasting temperature and peak viscosity
- Future – Distilling and Wine Making Capabilities

Sensory Program

- 8 sensory booths, Red or incandescent lights
- Difference Testing, Descriptive Round Table, Free Choice Profiling, Overall liking, True to type
- Core trained panel (12 people) in basic beer faults (FlavorActiv)



List of possible GMP reference flavor standards for training:

#	GMP Flavor Standard
1	Acetaldehyde
2	Acetic
3	Alkaline
4	Astringent
5	Bitter
6	Bromophenol
7	Burnt
8	Butryic
9	Caramel
10	Catty
11	Chlorophenol
12	Diacetyl
13	DMS
14	Dry hay
15	Earthy
16	Ethyl Acetate
17	Ethyl Butyrate
18	Ethyl Hexanoate
19	Geraniol

#	GMP Flavor Standard
20	Grainy
21	H2S
22	Isoamyl Acetate
23	Isovaleric
24	Kettle hop
25	Leathery
26	Lightstruck
27	Malty
28	Mercaptan
29	Metallic
30	Musty
31	Papery
32	Phenolic
33	Smoky
34	Sour
35	Sulphitic
36	Sweet
37	Worty
38	Yeasty

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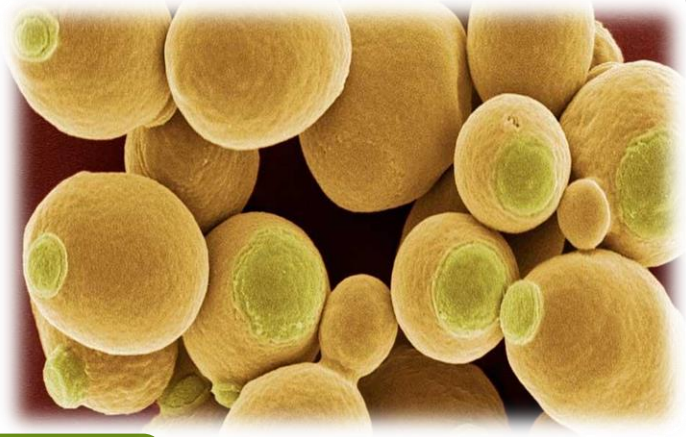
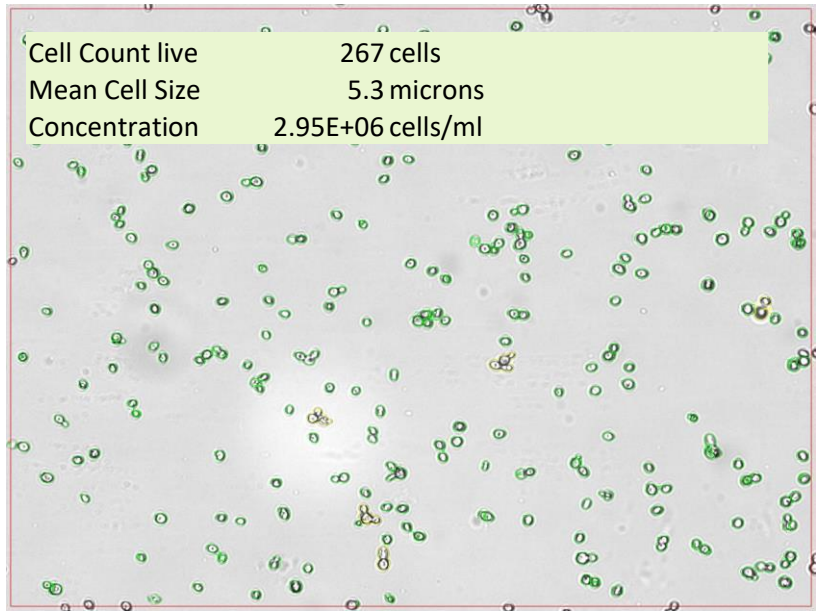
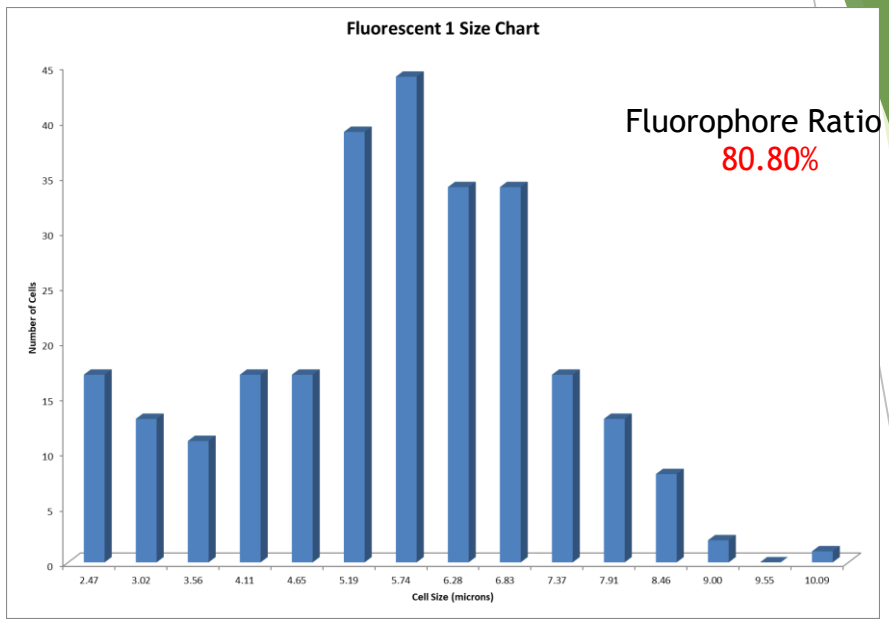
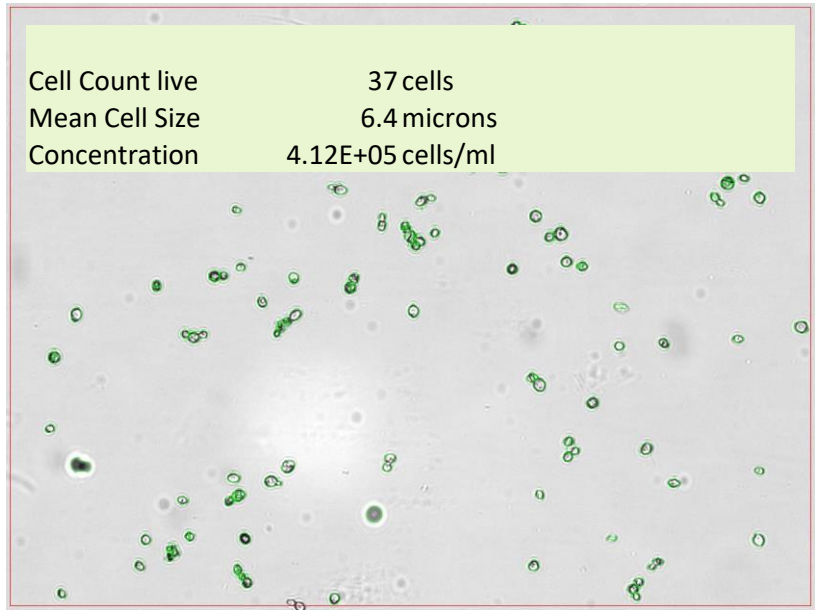


Yeast Vitality Change with Cycle Numbers

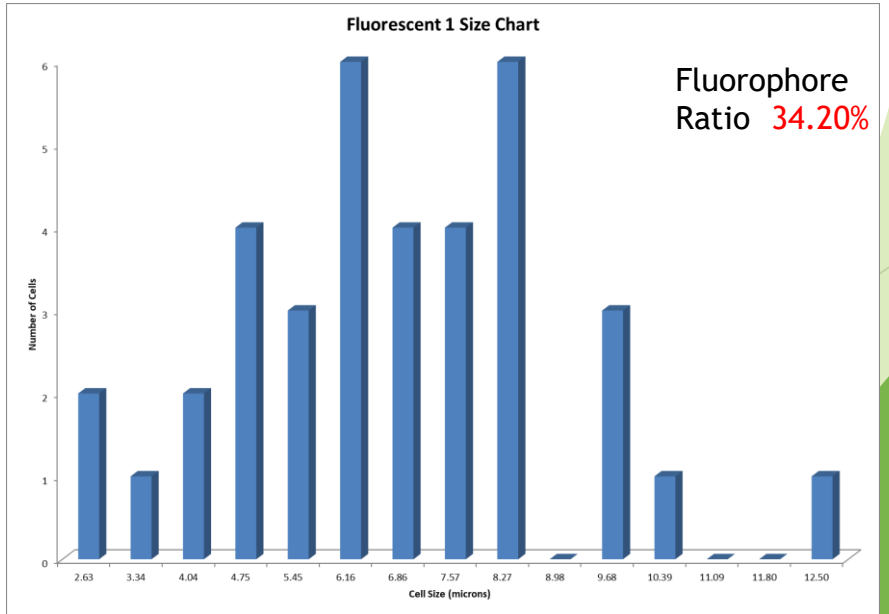


Gen 2 Yeast



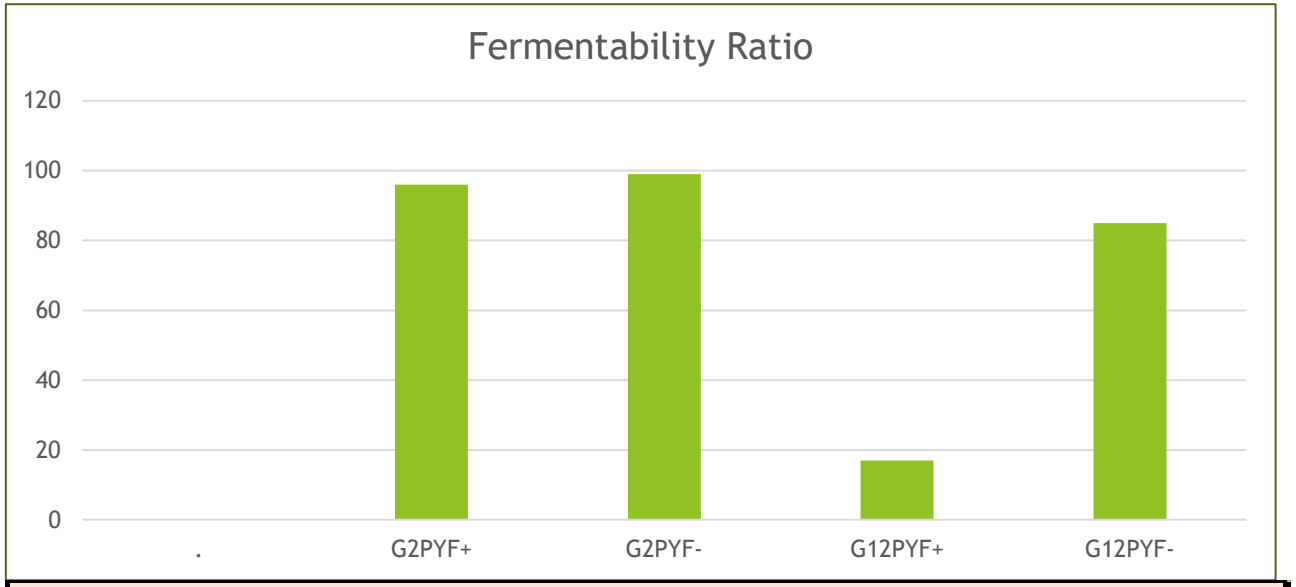


Gen 12 yeast



As yeast generations go up, vitality goes down

Case study - Rahr Tech Center
PYF (premature yeast flocculation)



MALT PYF	**+**	**-**	**+**	**-**
Yeast Gen	**2**	**2**	**12**	**12**
Beer Gravity fermented °P	**11.2**	**11.2**	**11 to 14**	**11 to 14**

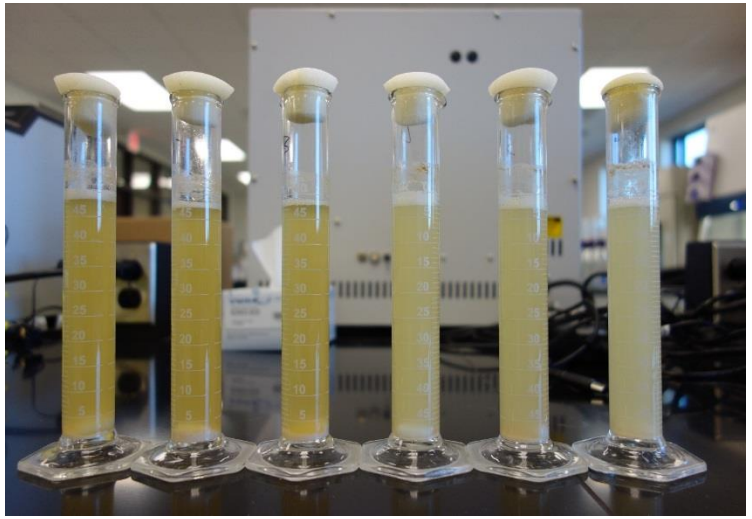
- Same strain of ale yeast but at different Gen./Cycle # responds to PYF malt very differently
- Higher generations demonstrated sensitivity towards PYF factors
- Ale strain can become PYF-sensitive

PYF Method Optimization at Rahr

-Brown, Aron, Yin & Kramer, 2017

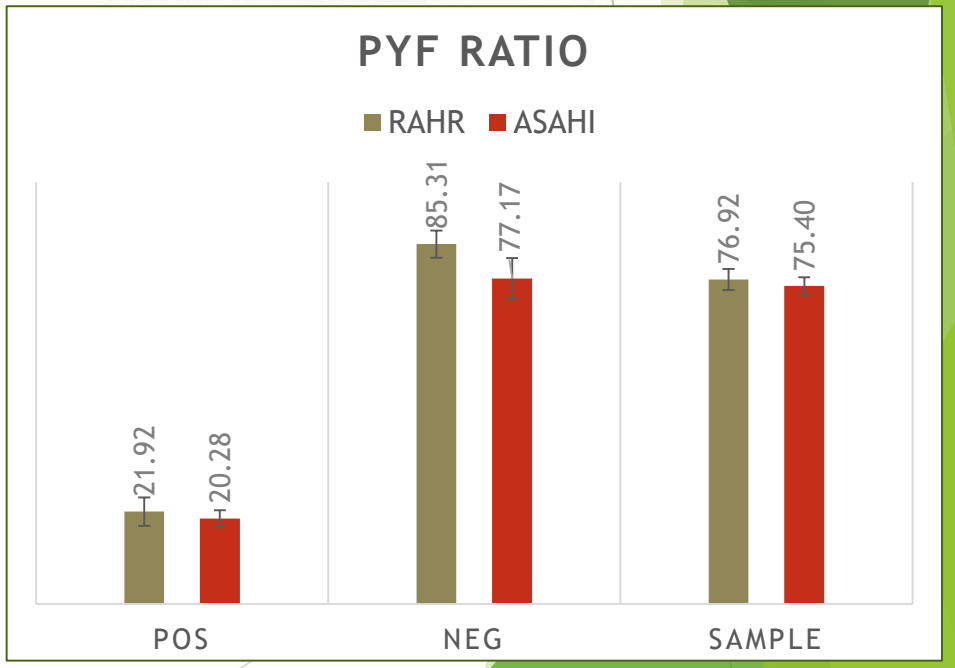
Practical Values

shorter time, smaller sample, and less risks in results



-8 day Method (Rahr)
-EBC tubes (600 ml)

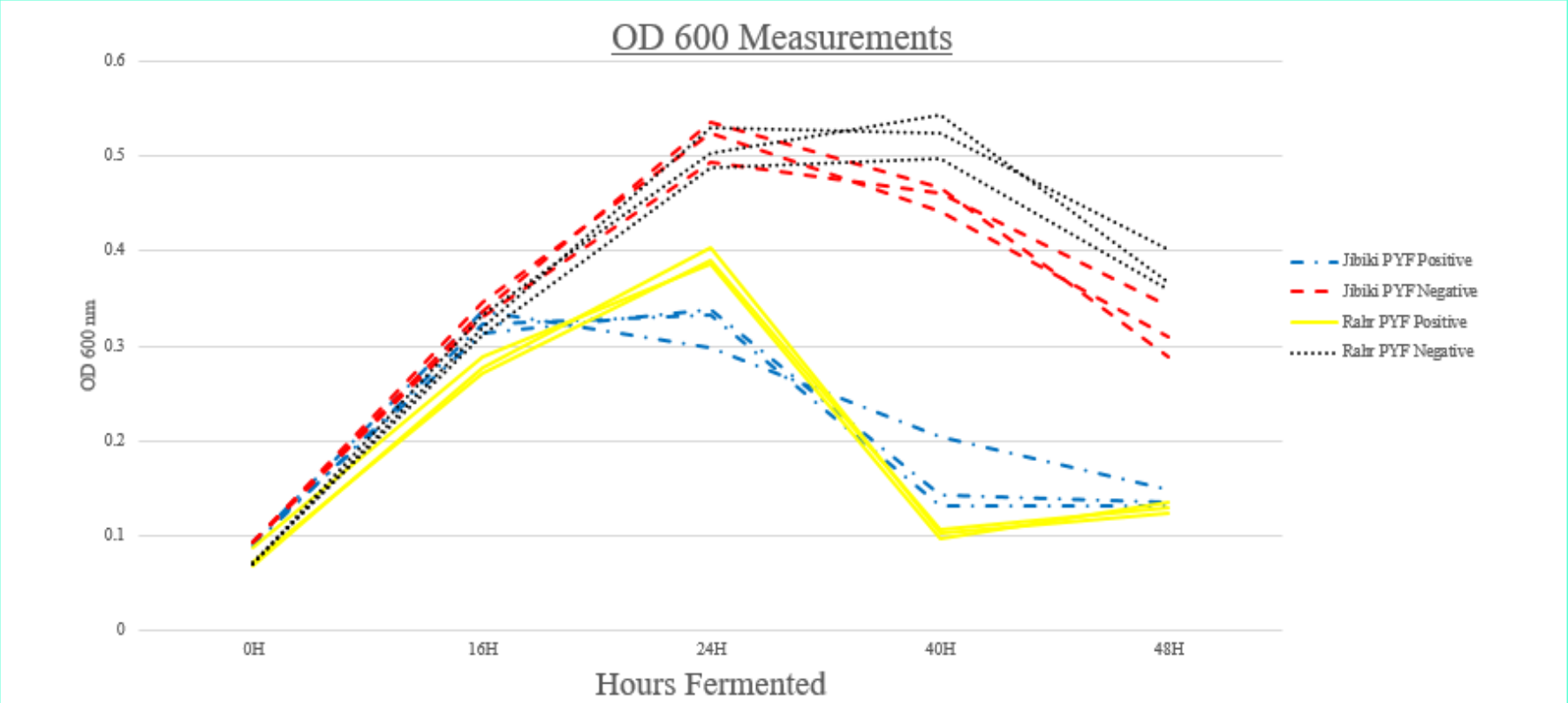
-Jibiki (Asahi ,2006) Method ~3 days
-50 mL graduated cylinder



Fermentation - Method Comparison

-Brown, Aron, Yin & Kramer, 2017

The graph below shows OD600 readings for PYF positive and negative samples pitched using yeast from the Jibiki et al. (2006) and the same samples pitched using yeast from the Rahr method.

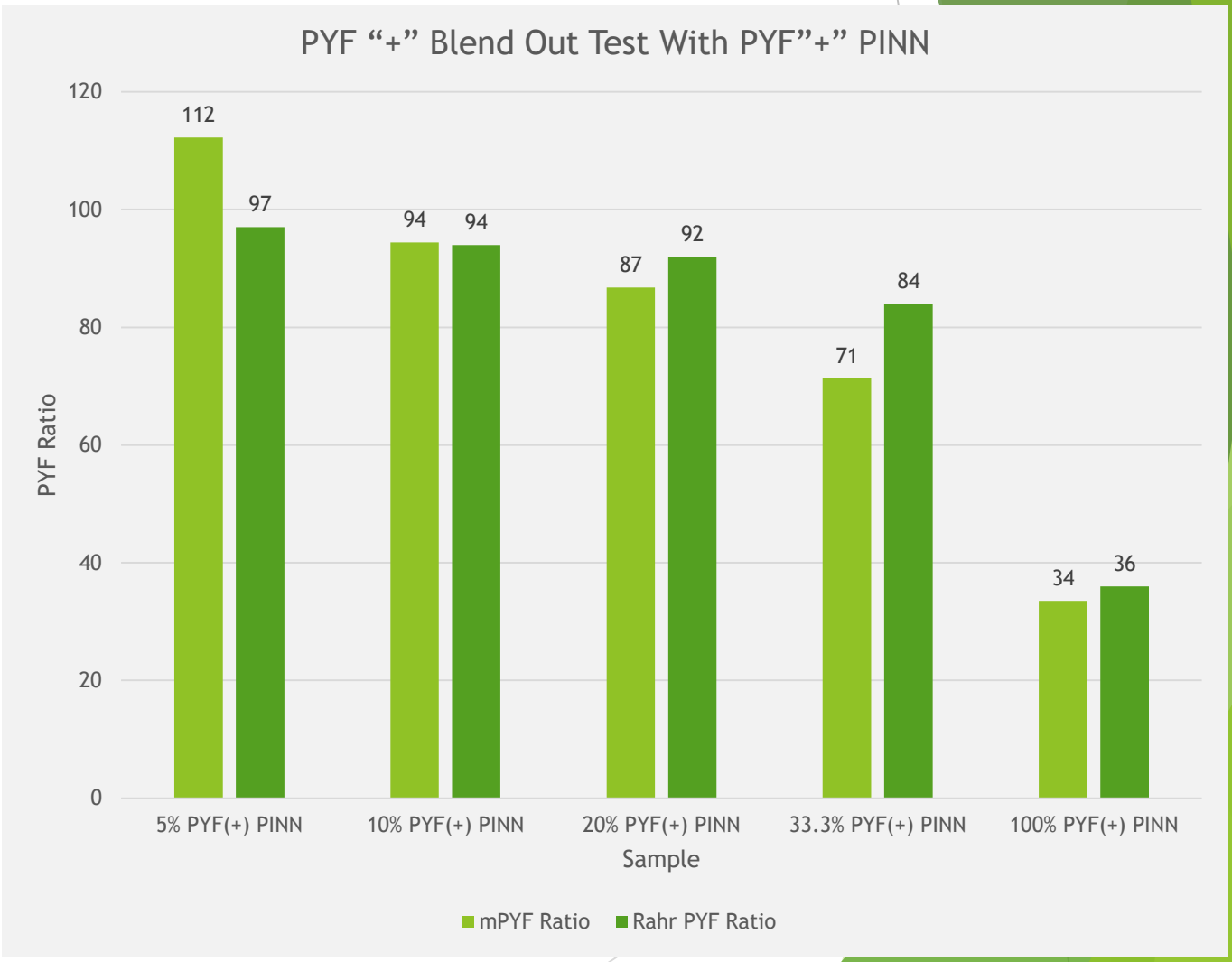


$$PYF\ Ratio = \frac{40\ H\ Cell\ Count}{24\ H\ Cell\ Count} \times 100$$

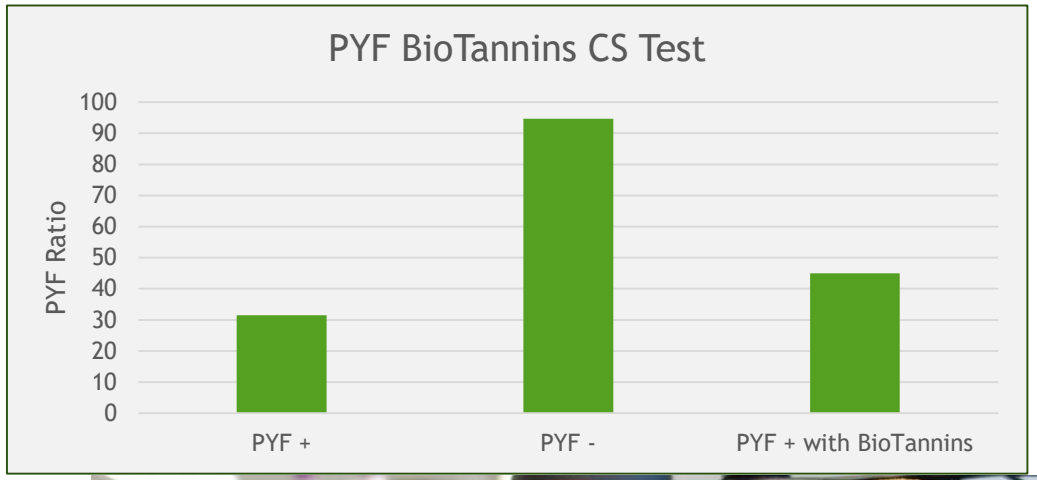
The PYF positive threshold is 40% and the PYF negative threshold is 80%.

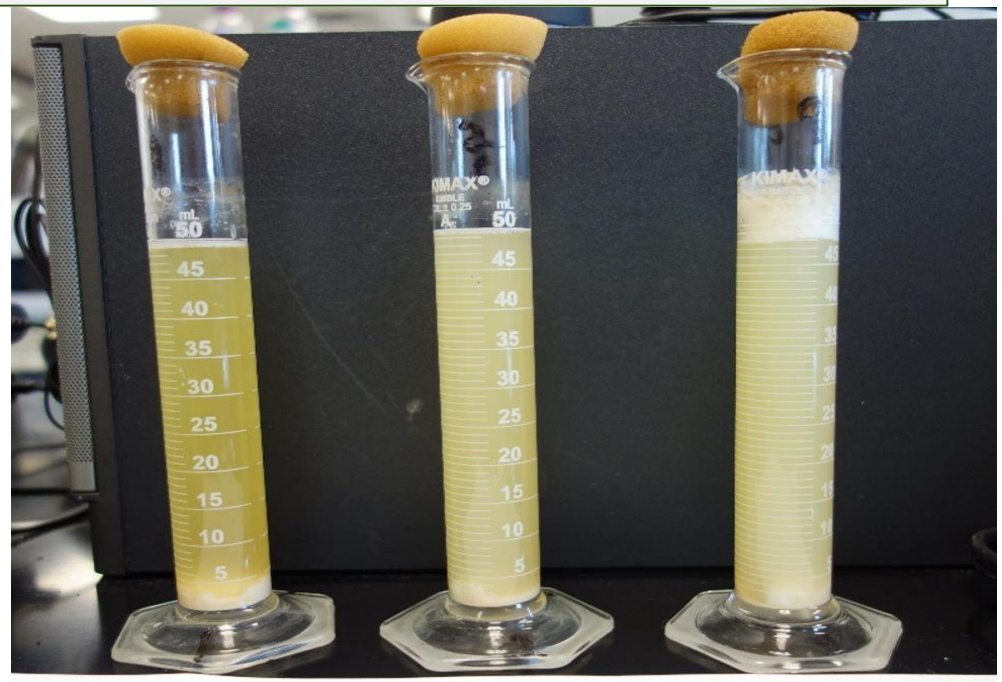
PYF Blend Test using a PYF-positive Pinnacle

- A PYF positive Pinnacle malt was blended with a PYF (-) control to determine at what point the malt will cross the PYF positive threshold, at the rate of 5, 10, 20, and 33.3%.
- The samples were shown to be PYF positive when blended with more than 1/3 PYF positive malt.
- This test was performed using two different PYF methods, of which the Micro PYF (mPYF) method is more sensitive.



BioTannin CS Treatment for PYF

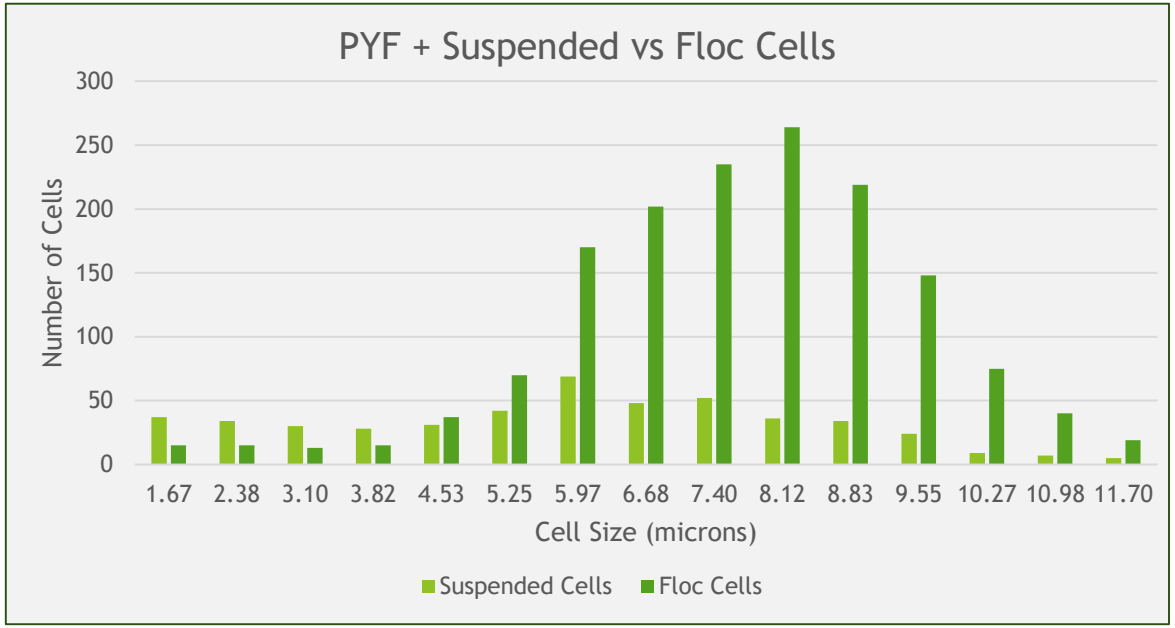


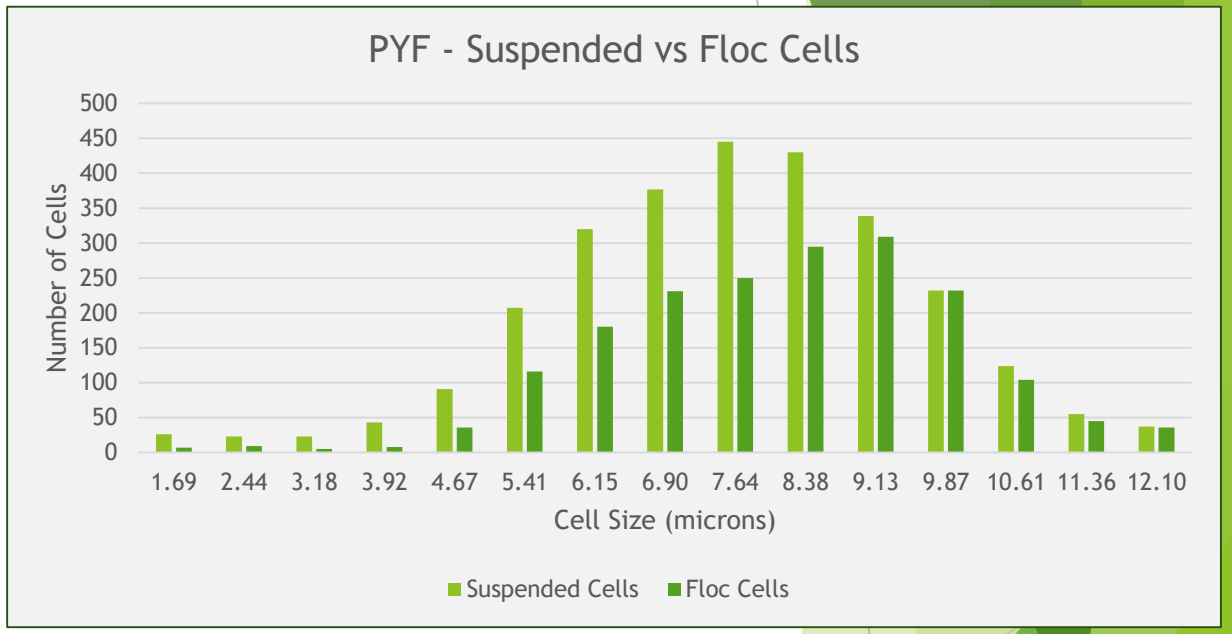


- Treatment for PYF was attempted by adding BioTannins to PYF + wort.
- Tripling the reported dosage to 6g/kg yielded minimal, but observable results.
- BioTannins not recommend to change the PYF factors at this time due to the following side-effects:
  - Significantly increased filtration times
  - Increased foaming (post filtration)
  - Possibly unwanted removal of proteins if added before filtration - Decreased foam stability of beer
  - SMA yeast was shown to flocculate both on the bottom and top.

Yeast Cell Size Distribution for Suspended and Flocculated Cells

- Significant difference was observed in average suspended yeast cell sizes of a PYF + control and PYF – samples at the end of our Micro PYF test.





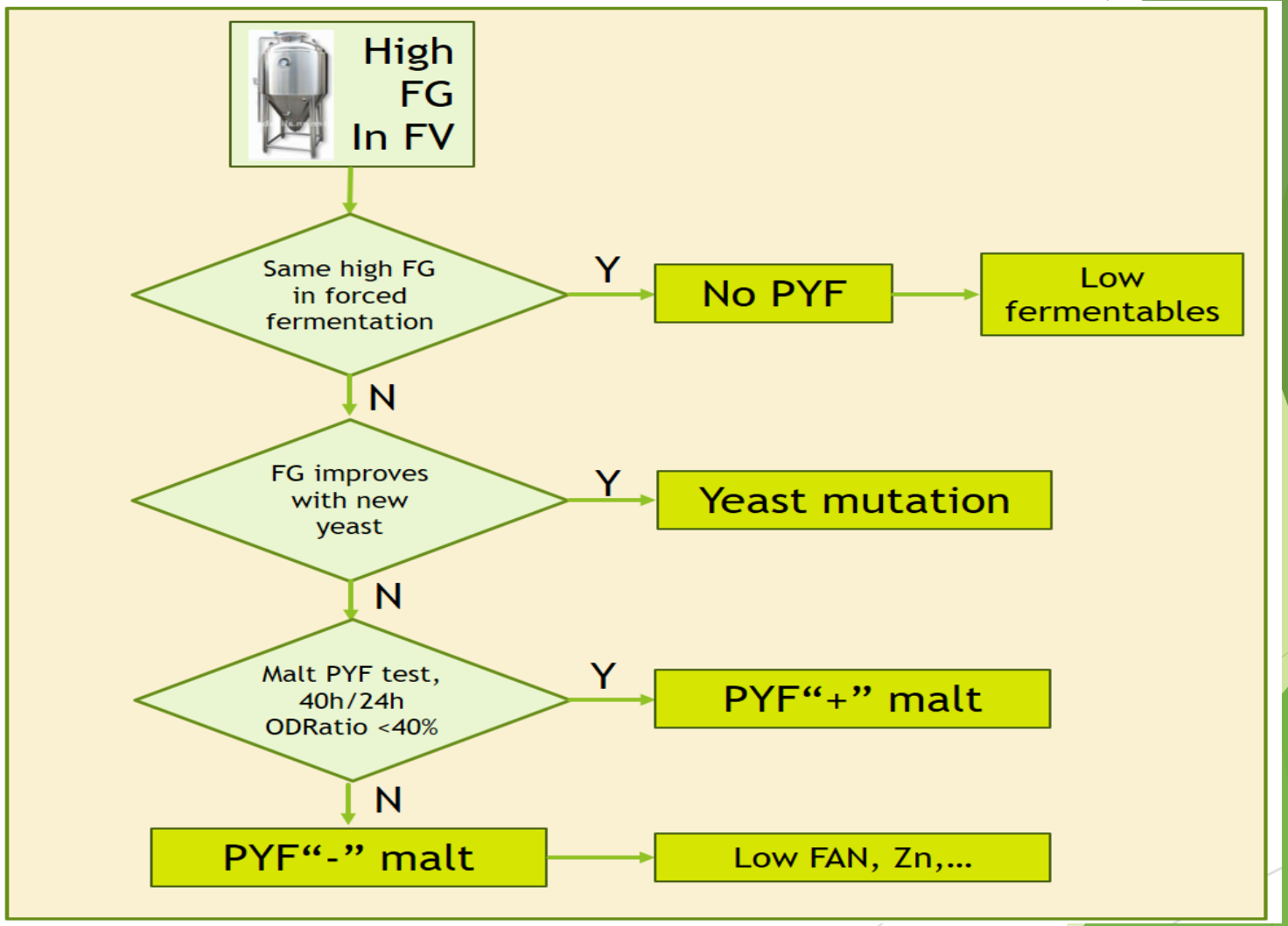
- Less yeast cells overall (in suspension and that have flocculated)
- Slightly bimodal distribution of cells in suspension
- Higher amount of young (small) cells in suspension

- More total cells than PYF + sample
- More normal distribution, slightly right-skewed
- Higher amount (proportionally)of larger (older) cells in suspension and of those that flocculated

Practical Approach to PYF Diagnosis

Potential root causes for high final gravity:

- Yeast mutation leading to PYF-sensitive heterogeneous culture
- Malt with PYF factor
- Deficiency in yeast nutrients like FAN, Zn…
- Low fermentable sugars in wort



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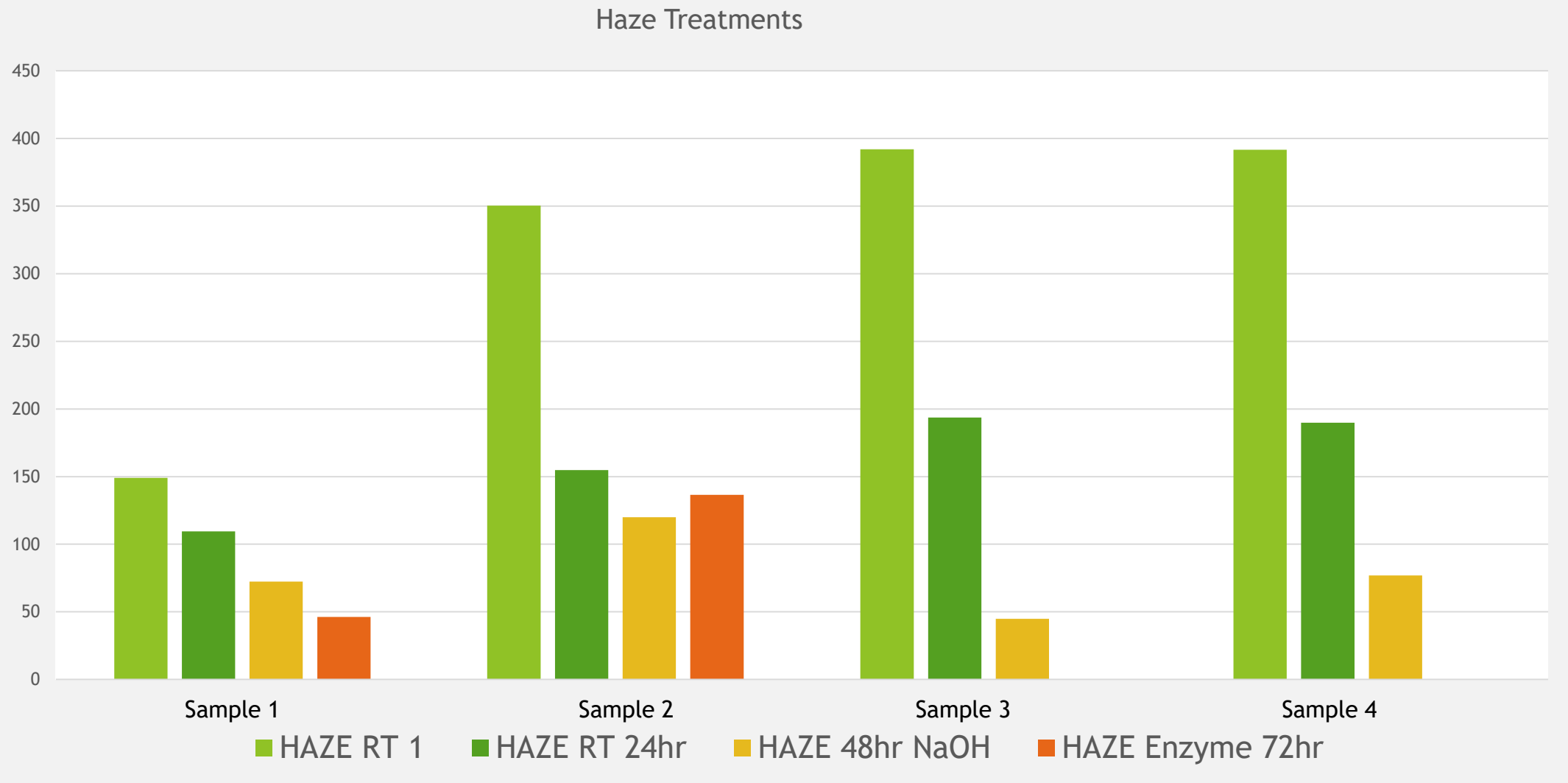
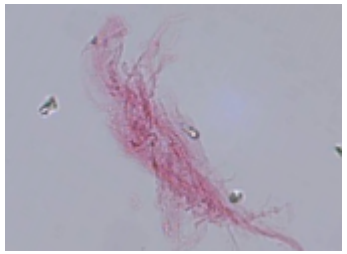
Case Study – IPA Haze Characterization



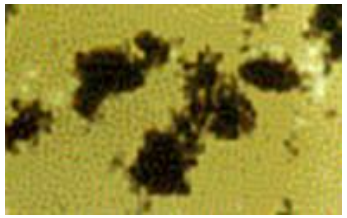
Haze Identification by Staining Techniques

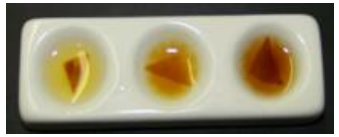
- Proteins (Eosin Yellow)



- Beta-glucans (Congo Red)

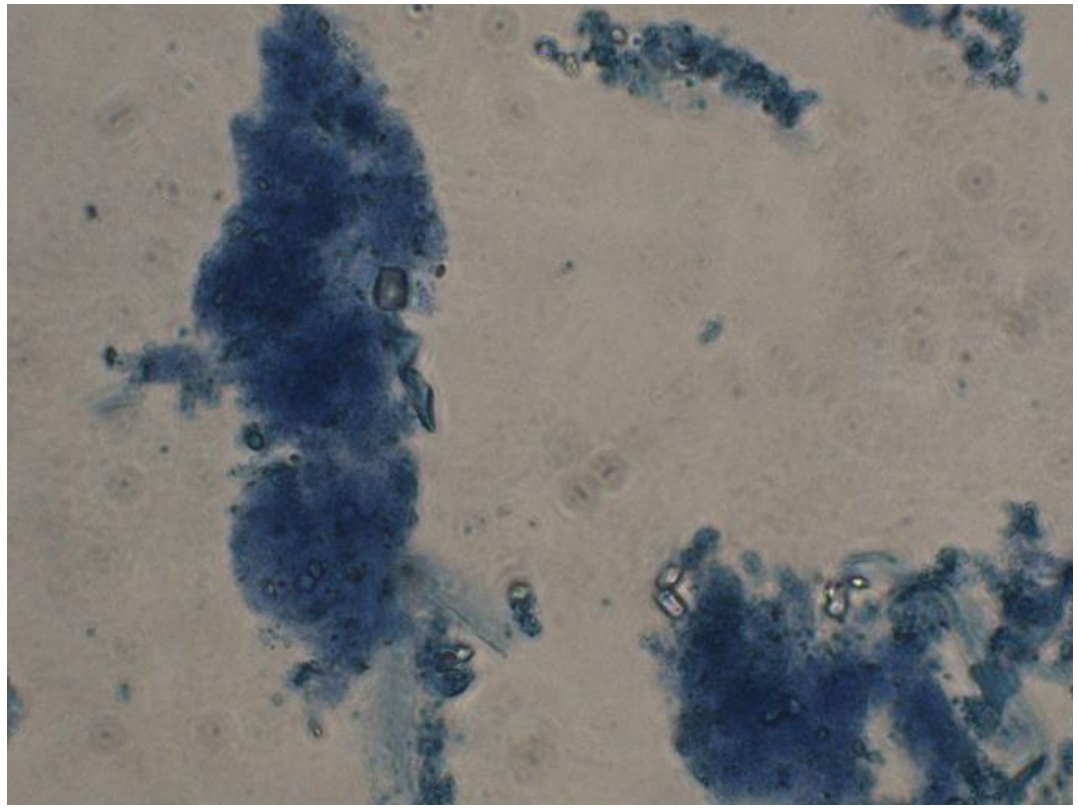
- Starch (Iodine/KI)



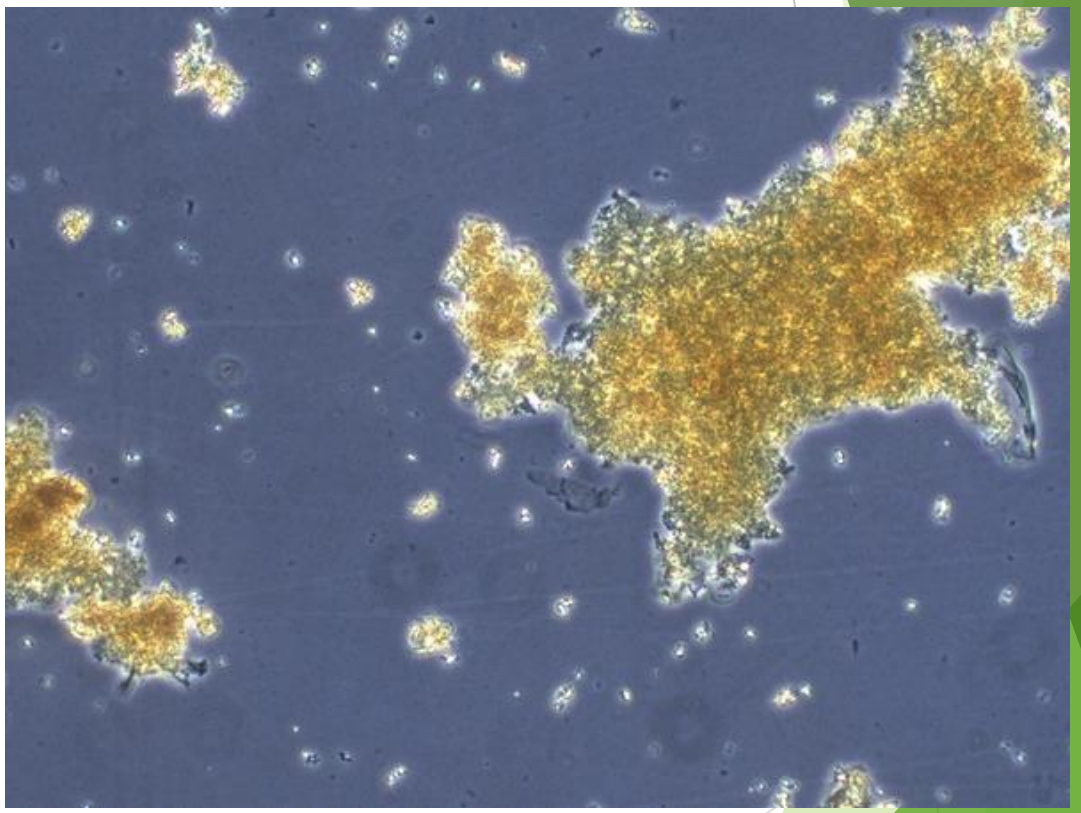


- Pentosans (Thionine)

Beer Haze investigation – Example

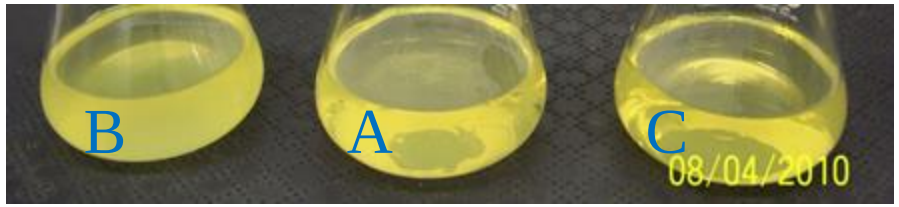


Calcium Oxalate in precipitate in IPA



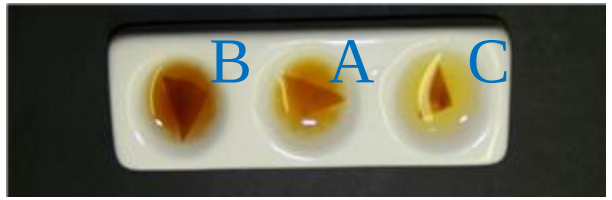
Dextrins in IPA sample

α -Glucan Characterization through iodide staining - an example of invisible haze

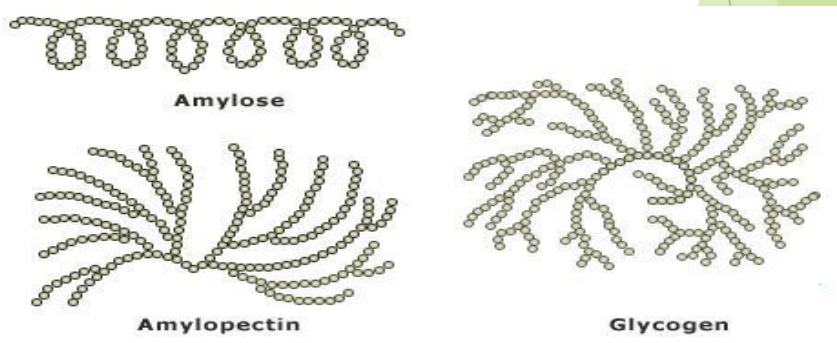


+ 2 vols EtOH, 48 hrs @4C

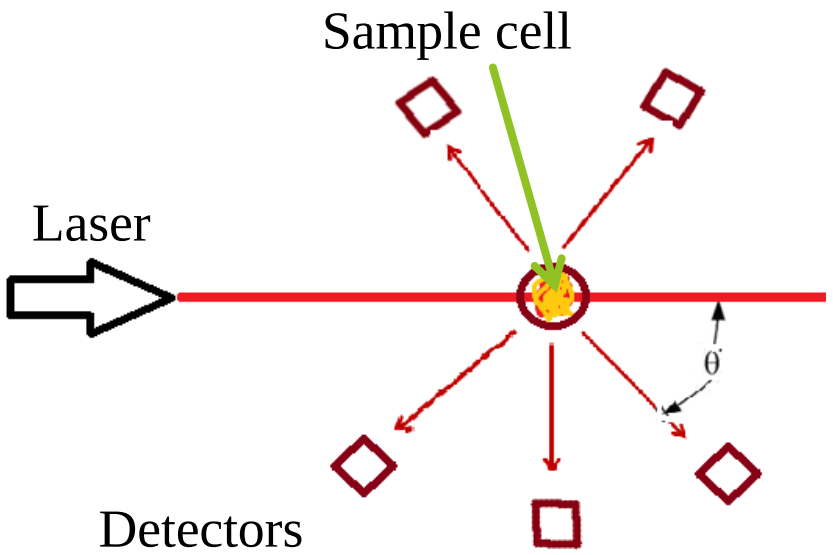
Caught and stained on 0.45µ filter paper

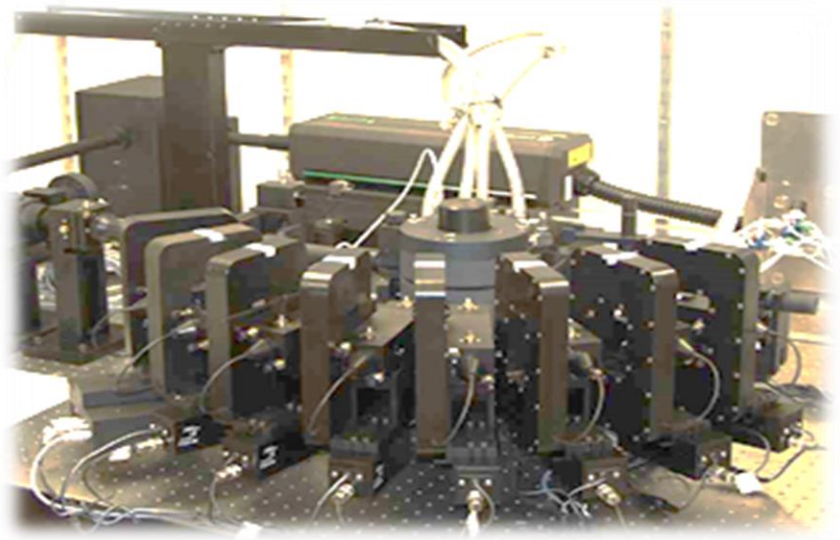


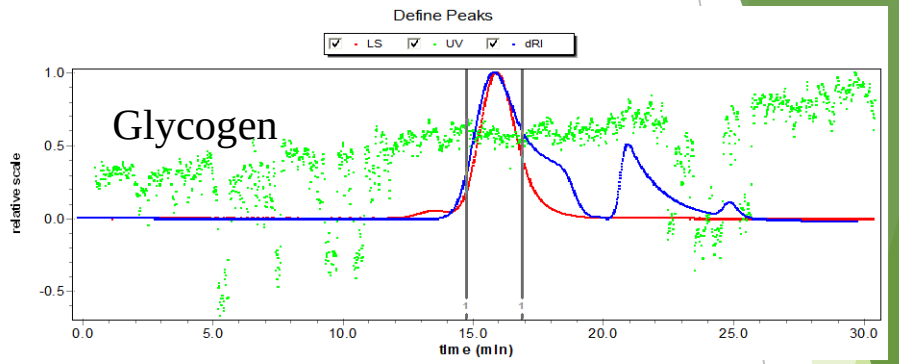
Beer B has more iodide staining power: amylopectin/glycogen

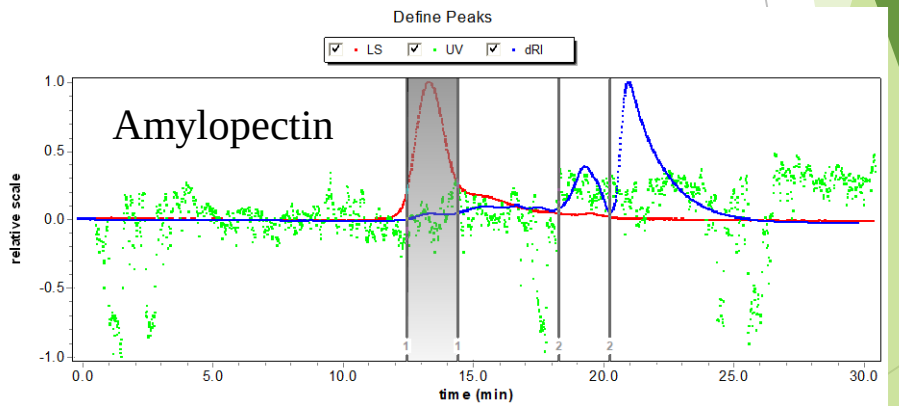


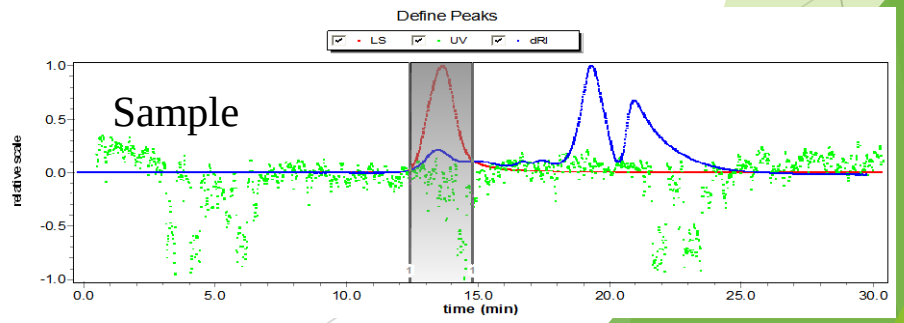
MALS- Multi-Angle Laser Light Scattering











Step-wise approaches for beer haze investigation

Hazy Beer

Clear @>20°C — Yes → Chill haze

No

Clear with NaOH drops — Yes → Permanent haze

No

Sediment by centrifuge /0.22 filter — Yes → *Staining identification → Starch, b-glucan, arabinoxylan etc

*Staining identification → Oxalate/DE, acidic polysacc. etc

No

**Enzymatic identification — Yes → Mannan(yeast), peptides, etc

No

Instrumental -MALS etc — Yes → Glycogen (yeast) or amylopectin (barley)

Data Exam
- Centrifugation speed
- Doses of finings
- Change in ingredient and processes

Process of elimination
- Change of beer pH: is it protein
- Change of temperature: chill haze?
- Does it filter out
- Tannoid/polyphenols

***Deposit staining**
- Proteins (Eosin Yellow)
- Beta-glucans (Congo Red)
- Starch (Iodine/KI)
- Pentosans (Thionine)
- Yeast/Oxalate/DE/Others

****Enzymatic identification**
- Amyloglucosidase (alpha-glucan)
- Hemicellulase (polysaccharides)
- Protease (Protein/peptide)
- Pullulanase (branched alpha-glucan)
- Iso-amylase (glycogen or amylopectin)
- Mannanase (mannan)

Instrumental_ MALS
- Glycogen – yeast storage carbohydrate
- Amylopectin – barley cell wall

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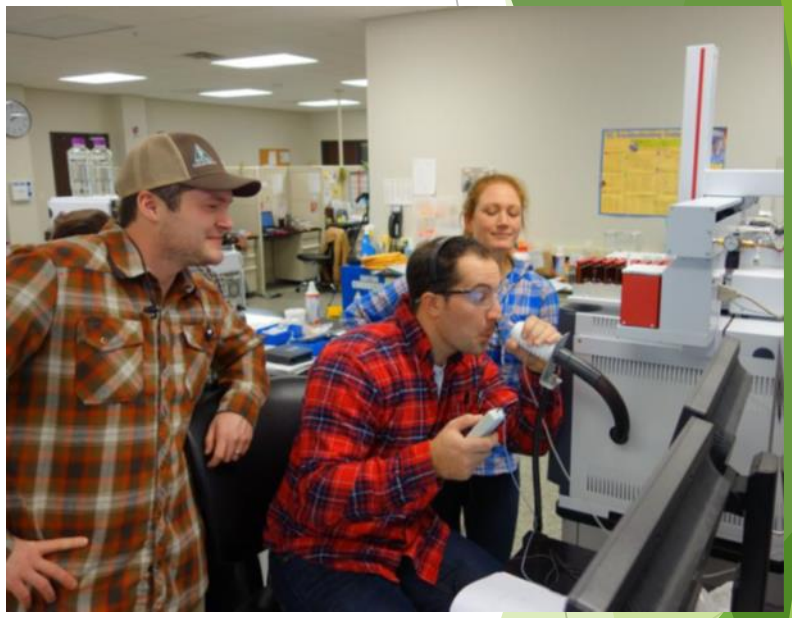
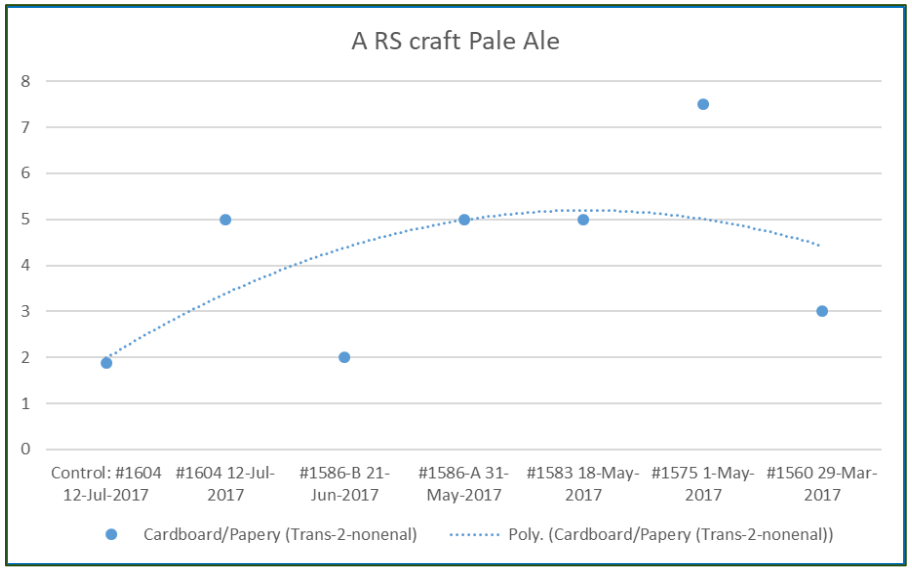
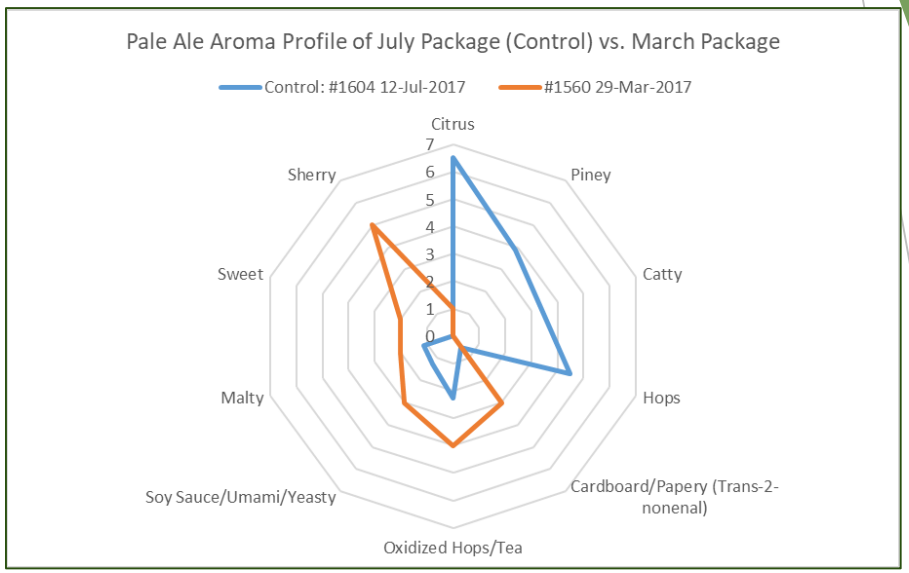
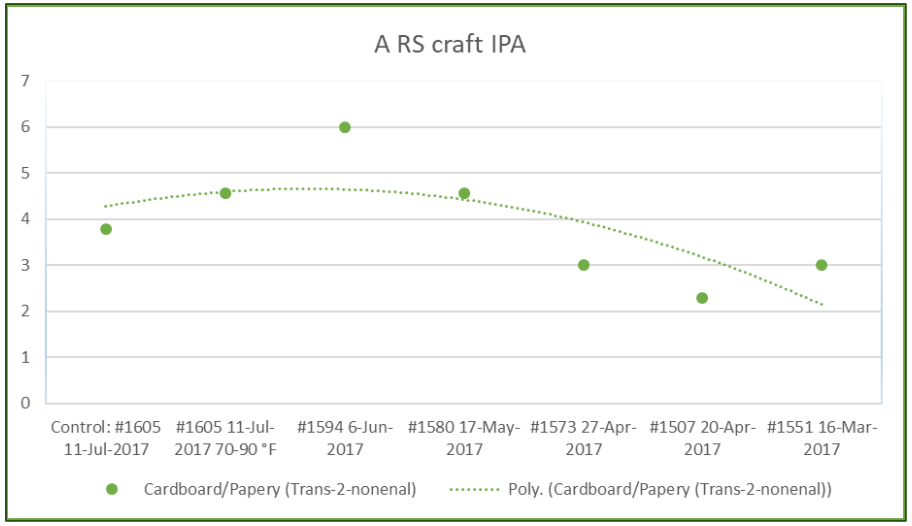
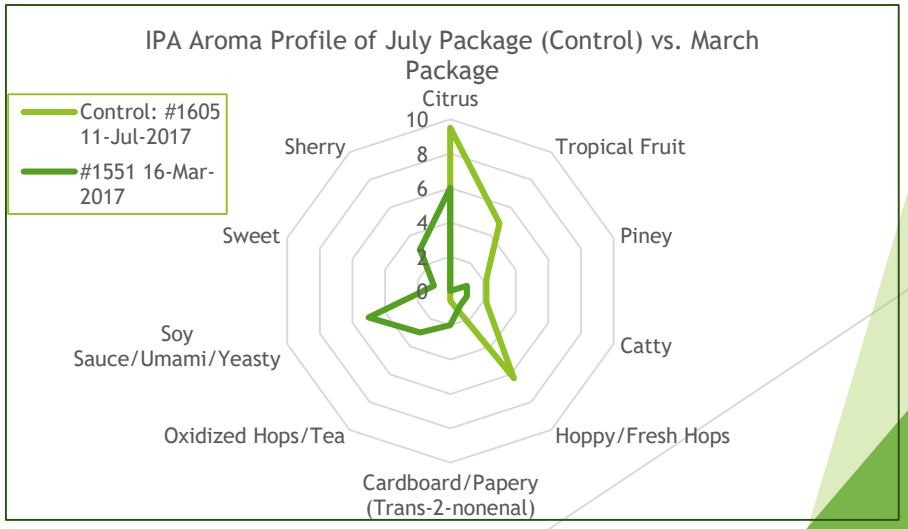


A case study of beer staling

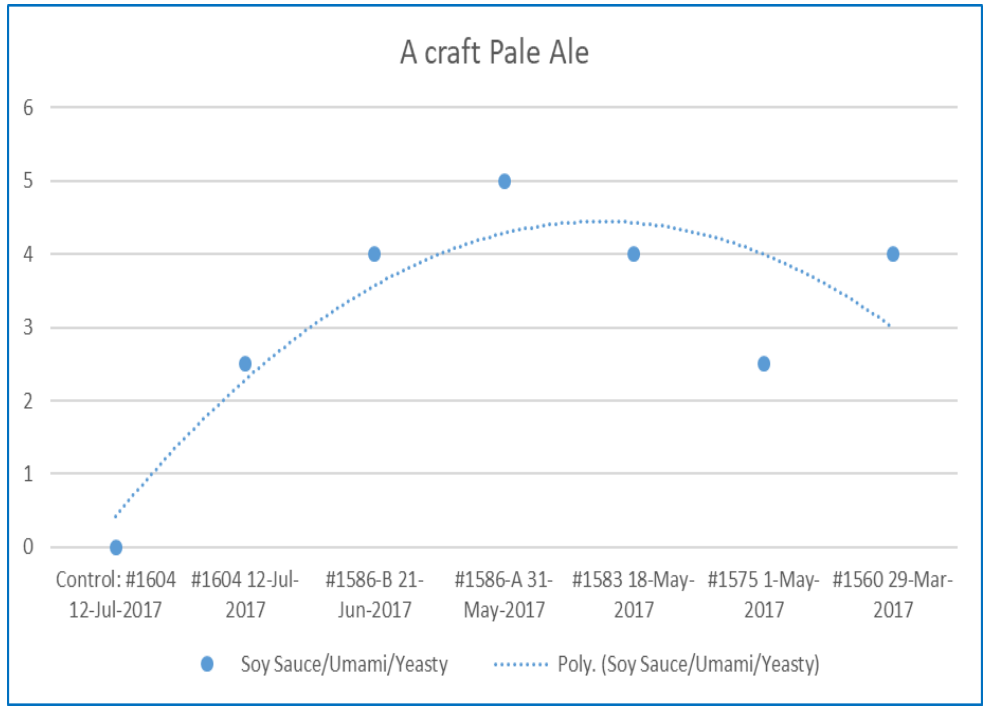


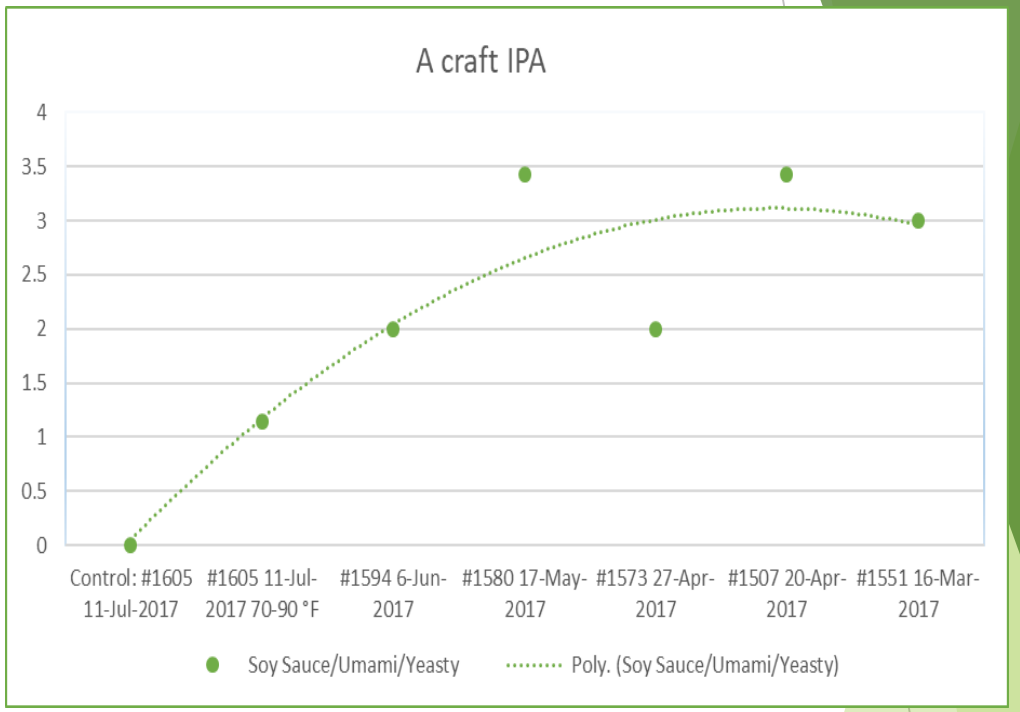






Flavor stability and beer analysis

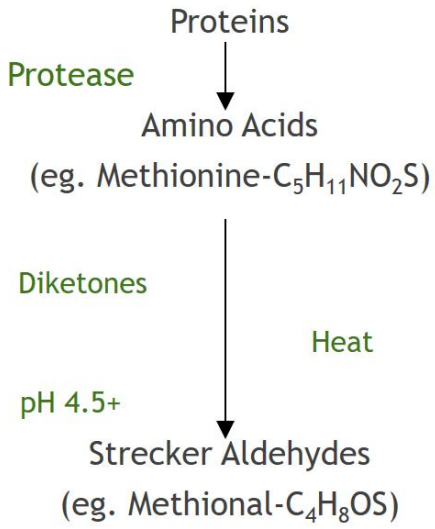




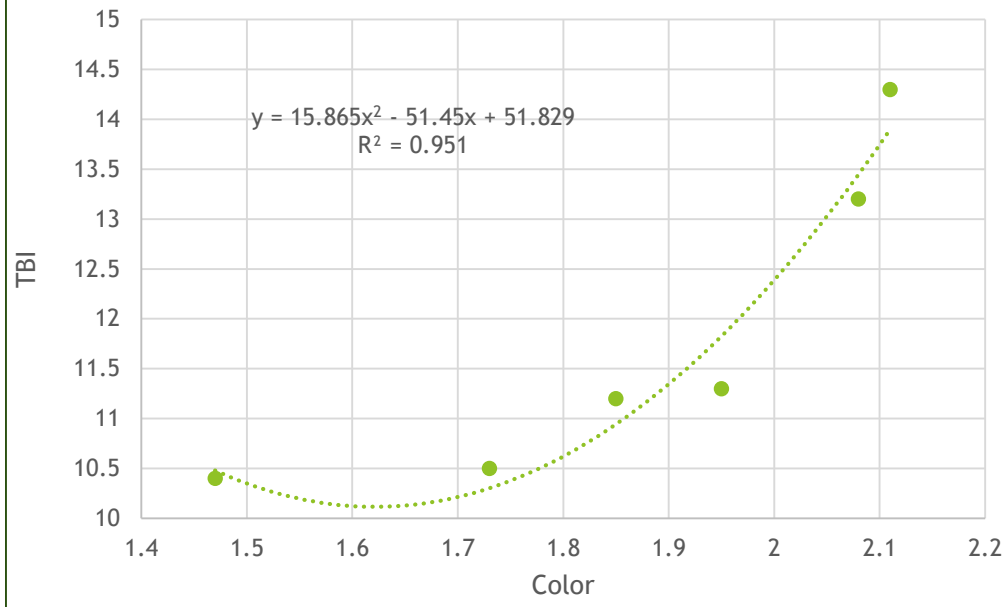
Sample	SG	° Plato	% ABV	pH	FAN (ppm)
Pale Ale (Control: #1604 12-Jul-2017)	1.010080	2.59	6.11	4.68	171
IPA (Control: #1605 11-Jul-2017)	1.010773	2.76	7.06	4.7	182

Staling factors: Strecker Aldehyde and Thermal Load

Strecker Degradation of Amino Acids

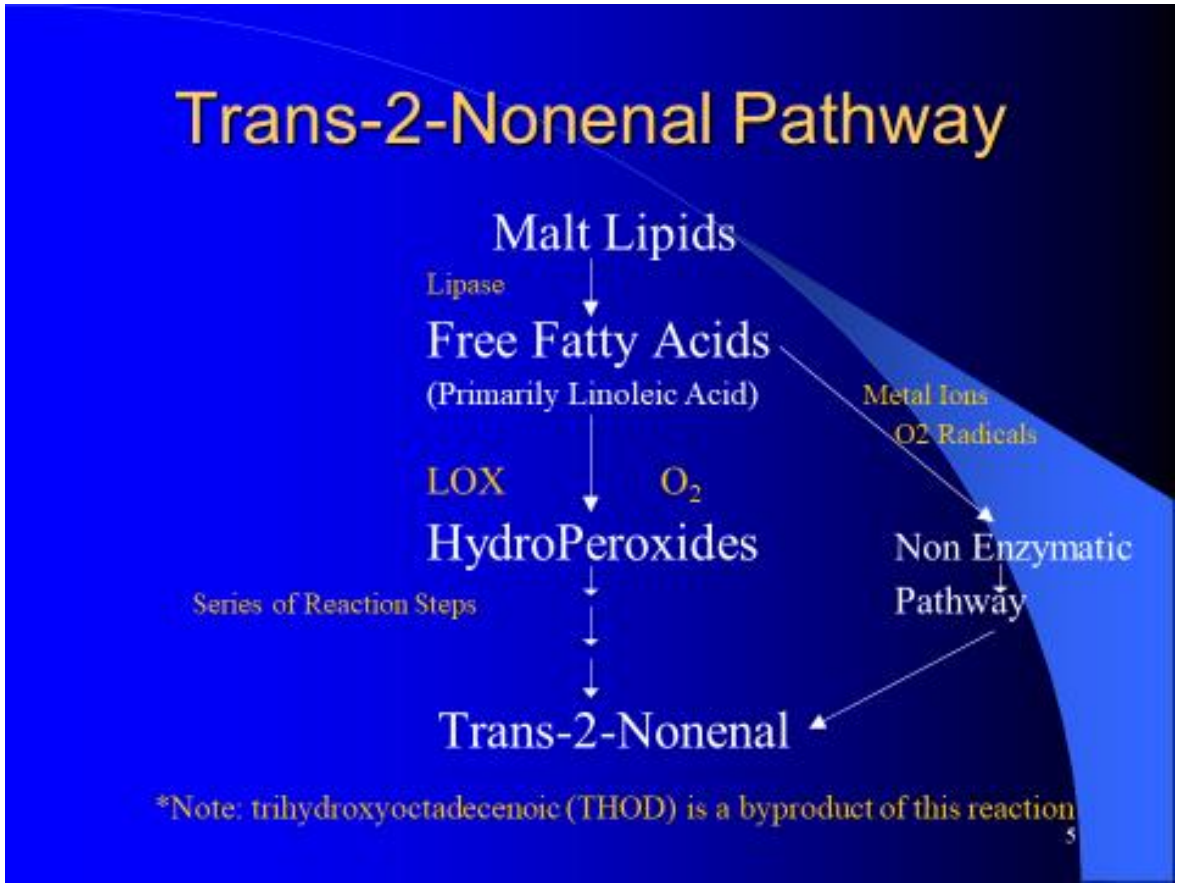


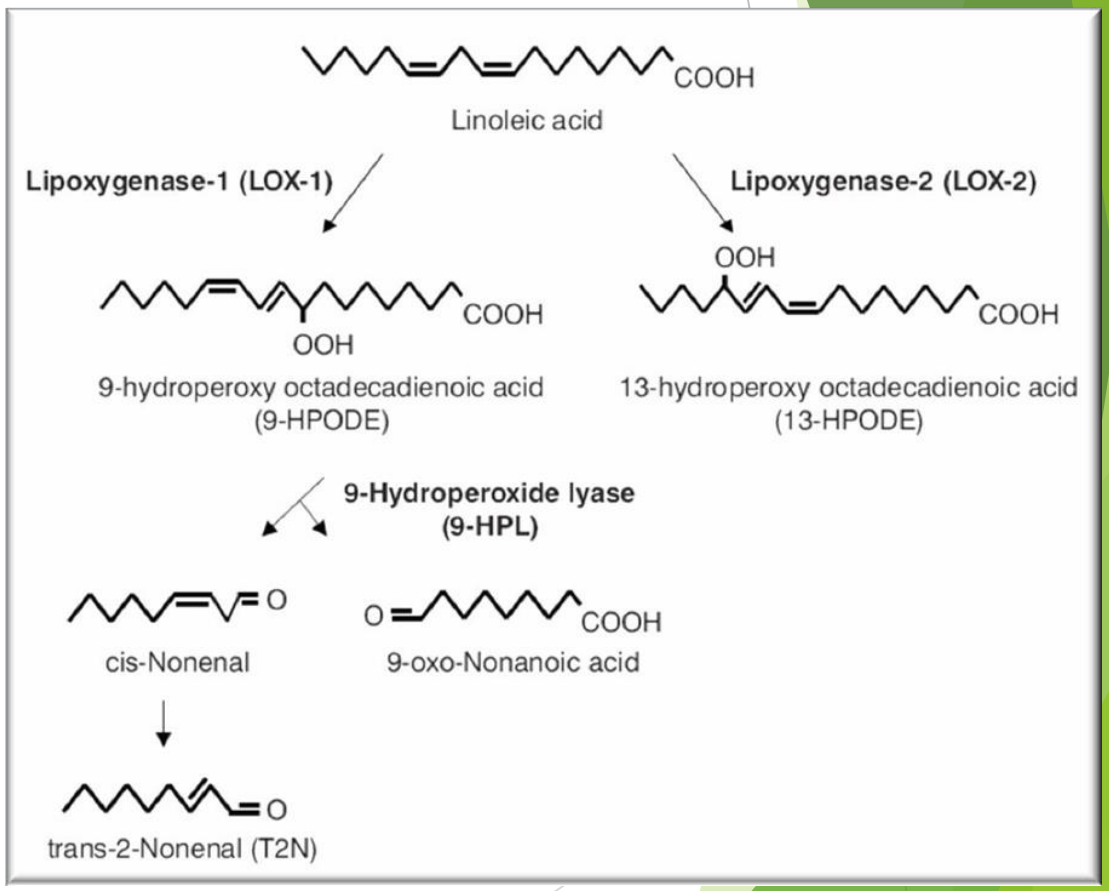
Color-TBI relationship by varieties



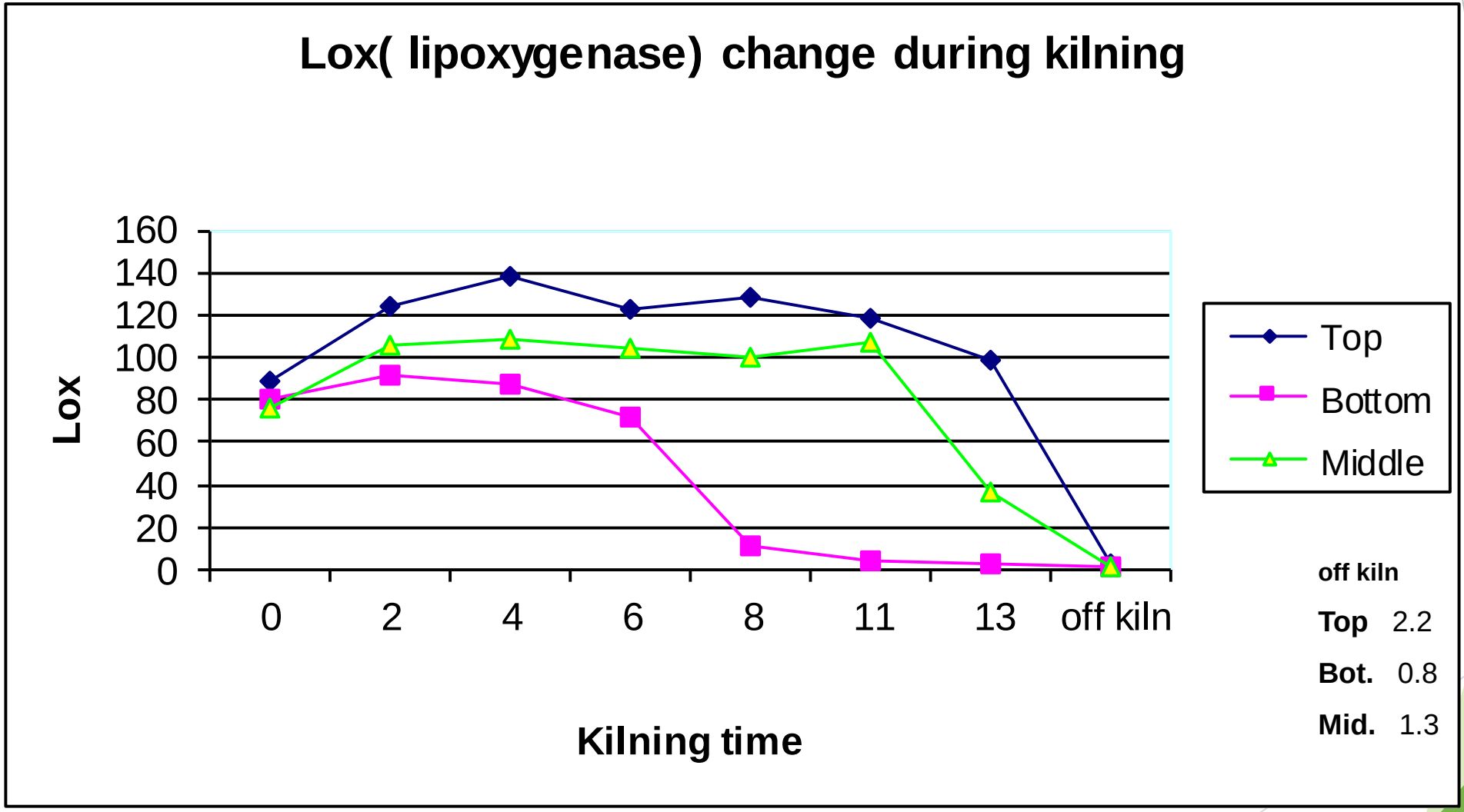
- Beer flavor stability is closely affected by malt, under complex barley and process conditions
- See separate report by Dr. P Aron for amino acid effect

LOX catalyzed cardboard/papery formation





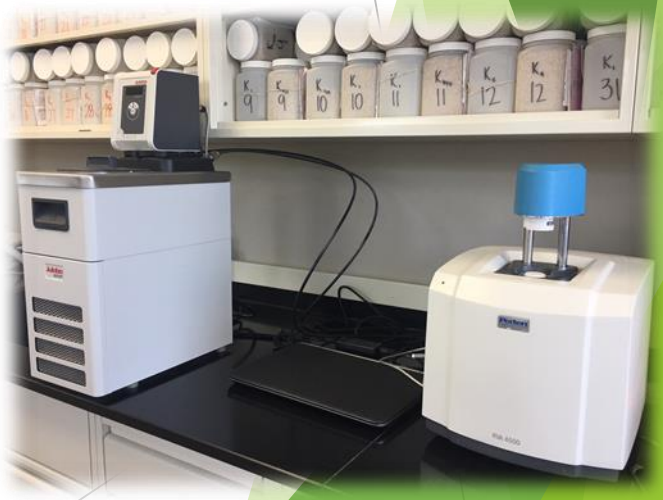
Control of LOX activities in malt kilning



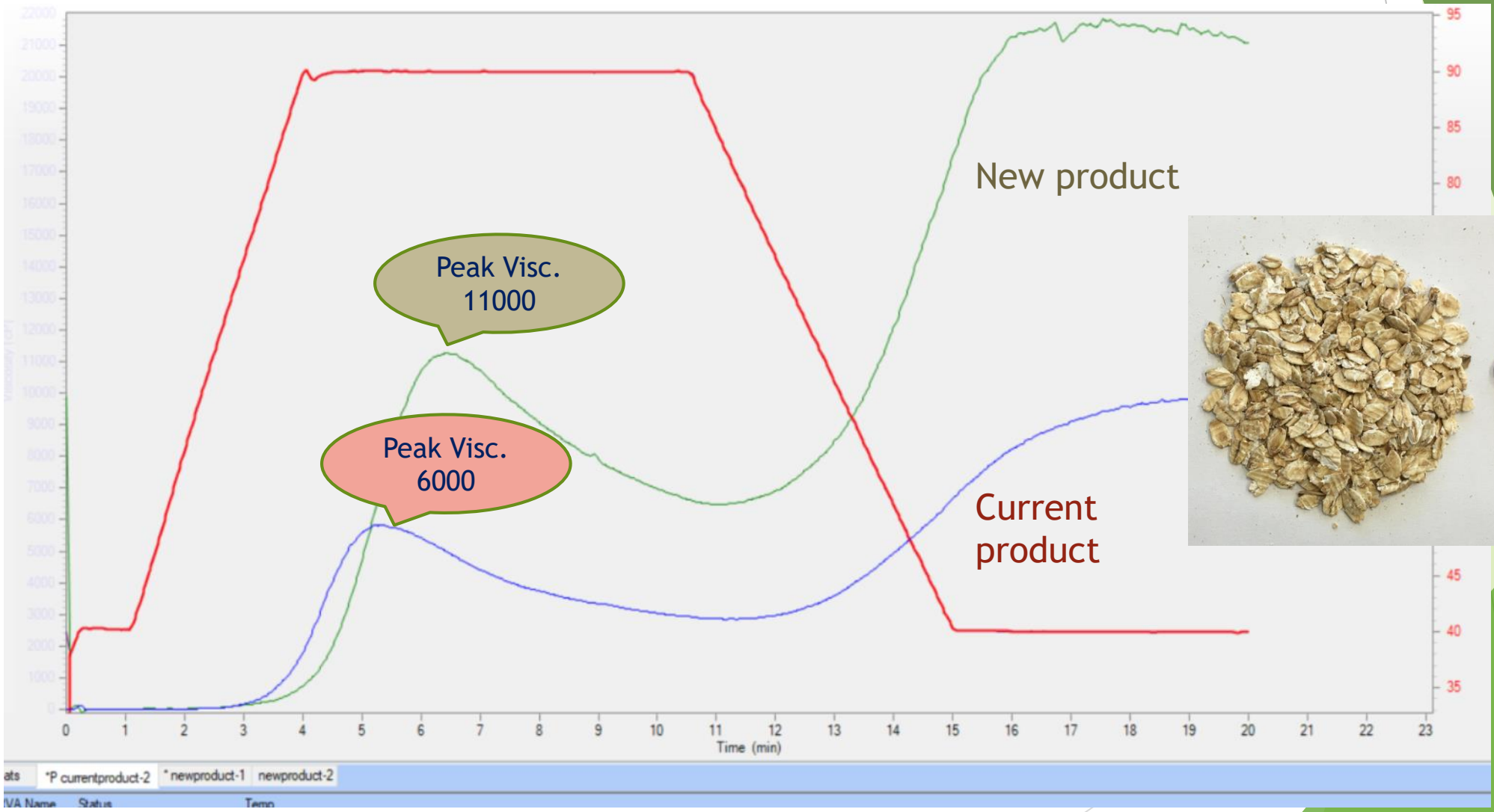
Yin, 2012

Agenda:

1. Introduction
2. Technical Capabilities
3. Case Studies
   - Yeast performance factors
   - Beer haze characterization
   - Beer flavor stability investigation
   - Starch gelatinization properties by RVA

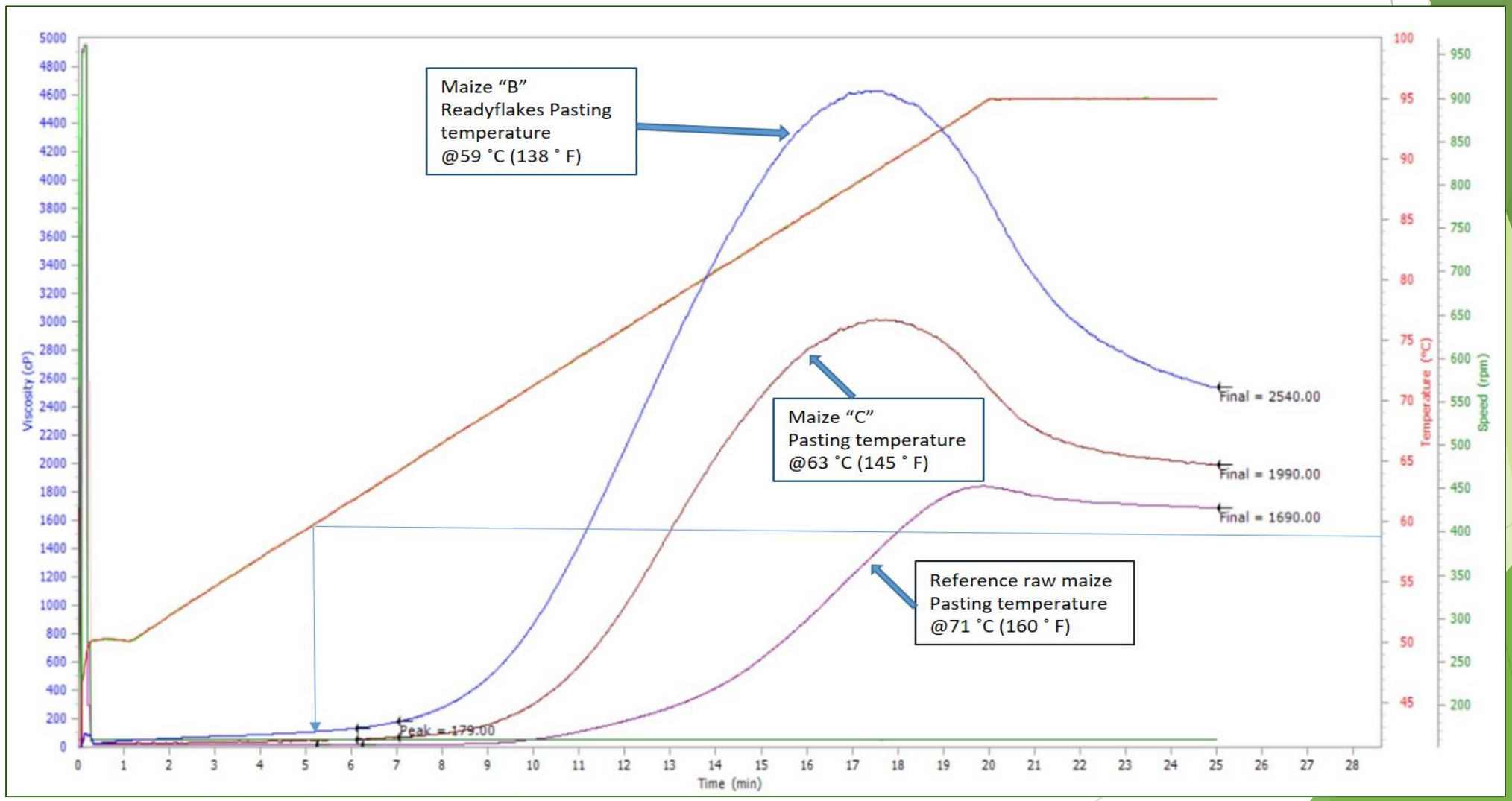


Flaked Oat - RVA comparison

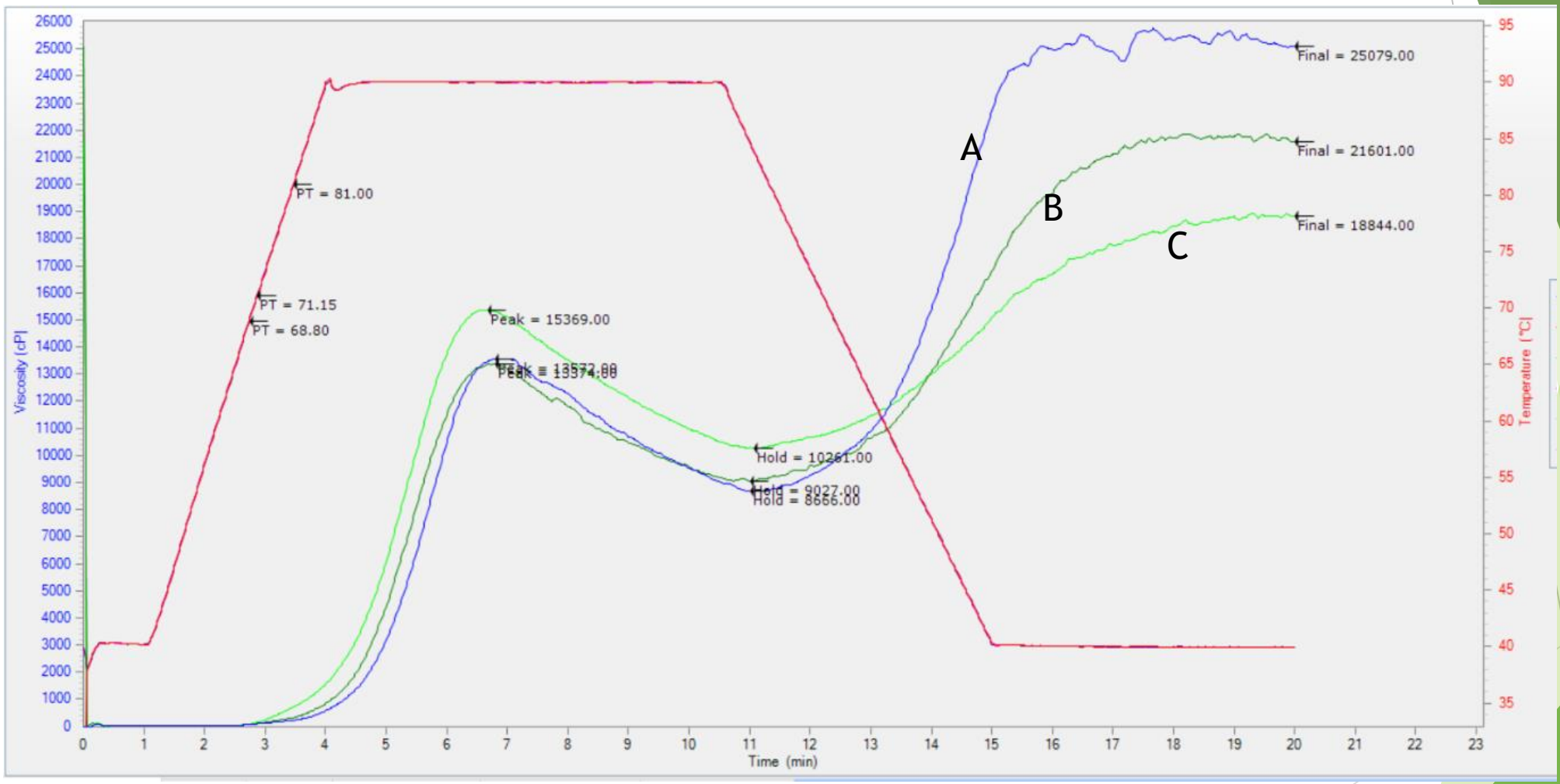


Corn grist RVA

- Gelatinization temp. variations decide point of grist addition



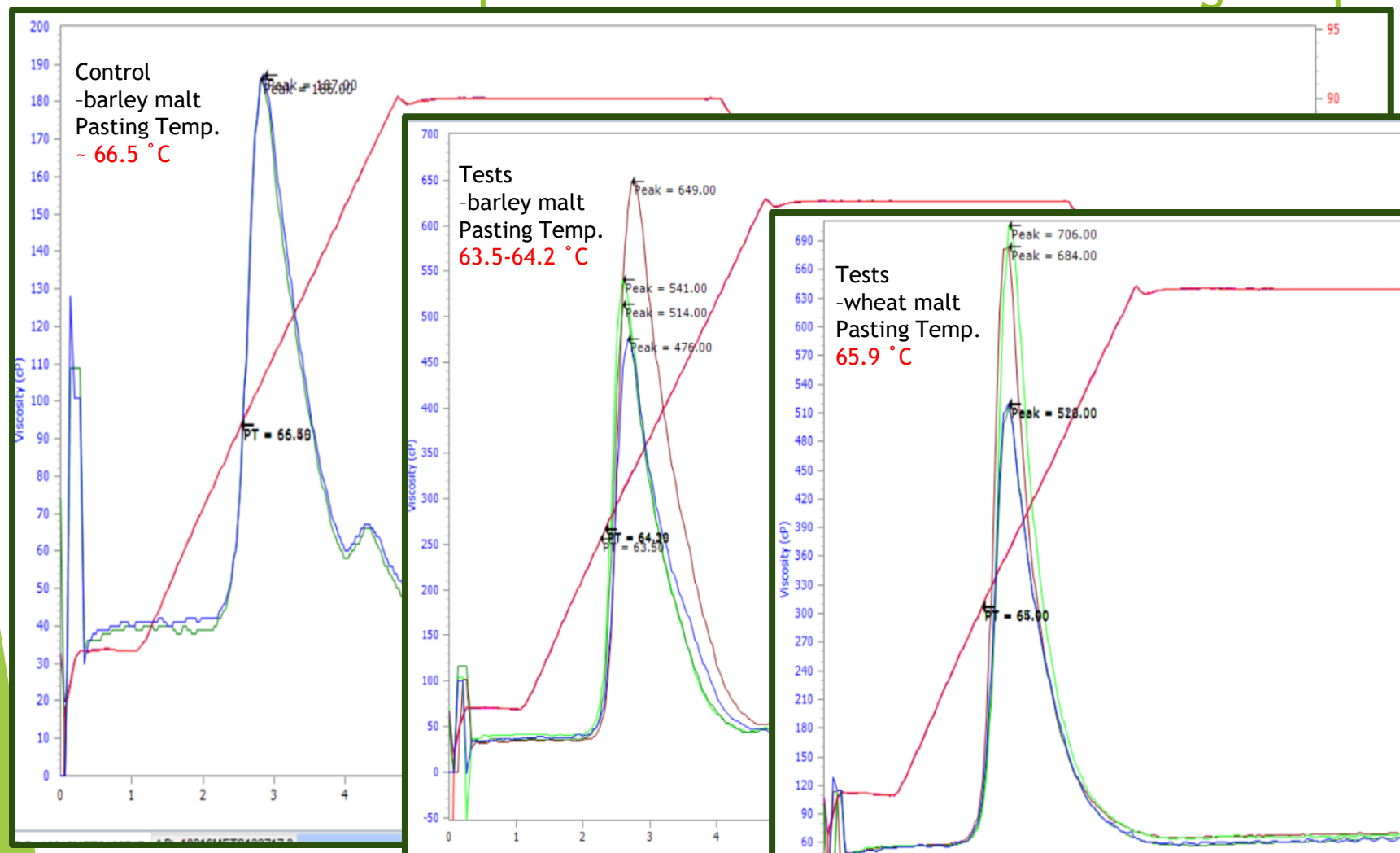
Oats flake RVA: for product optimization



- Extend pre-treatment to product A to lower the pasting temp ➔ Product C (81˚C => 69˚C).

Wheat and Barley malt RVA

- Gelatinization temperature variations ⟹ mashing temp. setting



Group A		Pasting Temp	Peak Visc.	Hold	Final
Malt 1	-1	64.2	408	62	68
	-2	64.3	418	58	66
Malt 2	-1	64.3	570	42	53
	-2	63.6	575	43	52
Malt 3	-1	64.4	411	35	60
	-2	64.3	407	36	59
Group B					
Malt 146	-1	67.3	241	37	40
	-2	67.2	235	36	38
Malt 241	-1	66.6	304	43	48
	-2	65.8	291	46	53
Malt 161	-1	66.5	175	37	40
	-2	67.2	170	37	40
Malt 226	-1	66.5	218	36	49
	-2	66.5	217	32	47

RAHR R&D standardized approach for technical solutions on raw materials, beer and brewing performance

Analysis

Malt

Hops

Yeast

Adjuncts

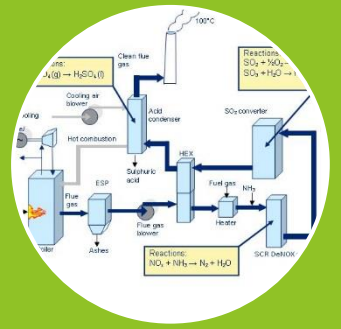
Beer

Sensory

Hot steep – Malt

Other ingredients

Beer



Processability

Malting

Brewing

Fermentation

Filtration

Scientific insight to brewing process and beer quality

Acknowledgements

- Rahr Tech Center R&D and QC teams
- BSG/Rahr Corporation

THANK ALL OF YOU!