# Dry-hopping Research at OSU
# A peek inside the Shellhammer Lab

Thomas H. Shellhammer
Department of Food Science and Technology

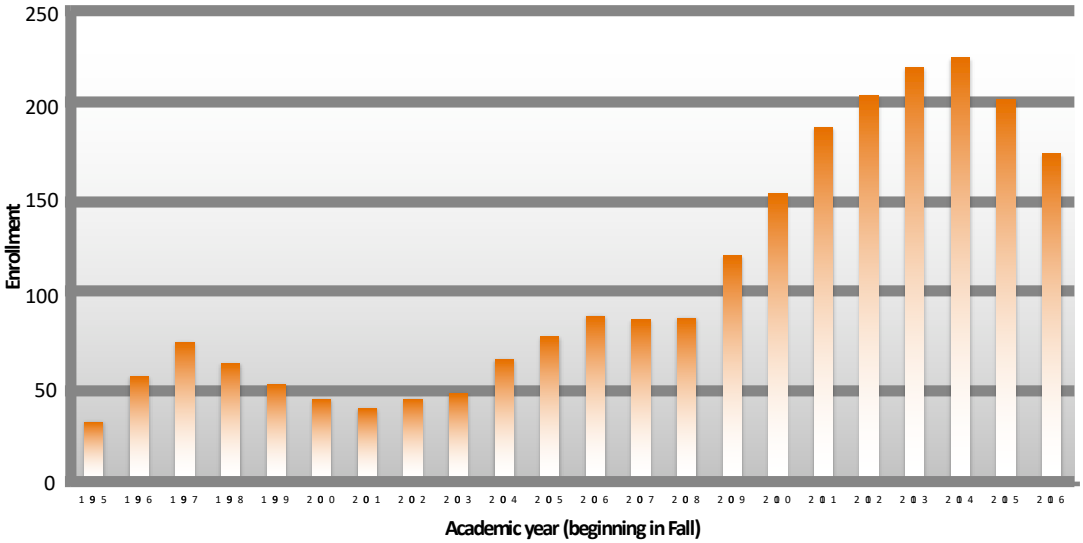
Oregon State University

## Brewing Science (and beer-related) at OSU

- **Brewing Chemistry/Engineering** – Tom Shellhammer
- **Brewing Micro/Genetics** – Chris Curtin
- **Pilot Research Brewery** – Jeff Clawson
- **Instructor/Advisor** – Glen Li

- **Barley breeding & malting** – Pat Hayes
- **Hops breeding** – Shaun Townsend & John Henning
- **Hops pathology** – Dave Gent
- **Hops & health** – Fred Stevens
- **Beer Economics** – Vic Tremblay
- **Oregon Hops and Beer Archives** – Tia Edmunson-Morten

# Undergraduate enrollment in FST




# Exciting times for brewing research and teaching

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## Dry-hopping research in the Shellhammer lab

- Whirlpool vs Dry-hopping – Cultivar differences
- Dosing rates and extraction efficiencies
- Bitterness pick up during dry-hopping
- Hop creep – overattenuation following dry-hopping
- Hop enzymes impact beer quality

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Daniel Sharp, Ph.D.
Director of Brewing Operations
Ninkasi Brewing Company

# WHIRLPOOL VS. DRY- HOPPING: SENSORY ANALYSIS

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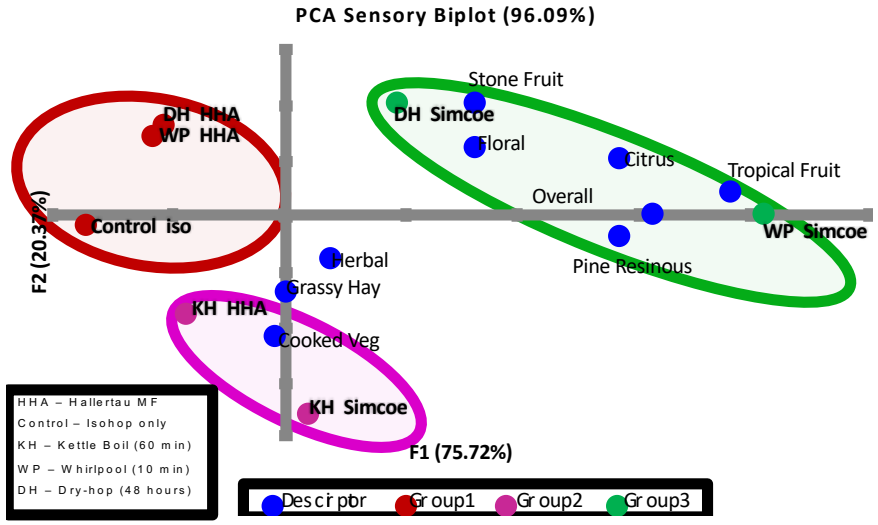
## Contributions of select hopping regimes to the terpenoid and aroma profile of ale and lager beers.




Daniel C. Sharp, Yanping Qian, Gina Shellhammer & Thomas H. Shellhammer (2018) Contributions of Select Hopping Regimes to the Terpenoid Content and Hop Aroma Profile of Ale and Lager Beers, Journal of the American Society of Brewing Chemists, 75:2, 93-100, DOI: 10.

# EXPERIMENTAL DESIGN

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# Experimental Design

**Objectives:**

- Minimize confounding variation
- Factors: Cultivar (12) and Addition (2)
  - Subfactors: KO order, Kettle boil, Fermentation variation

**Challenges:**

- Only 2 factors but total treatments = 24
- Complete design = 25 units *2 reps* 6 sensory reps = 300 samples.
- Brew length

# Experimental Setup

12 Cultivars

Amarillo
Cascade
Chinook
Citra
Halletauer Mittlefrüh
Huell Melon
Galaxy
Mosaic
Nelson Sauvin
Nugget
Simcoe
Saaz
Unhopped

Base

- 12°P wort
- 100% pale ale malt
- California ale (18°C)
- 25 ppm IAA

Hopping – 4 g/L (~1 lb BBL)

- Whirlpool: 25 min @ 87-95°C
- Dry-hopping: 72 hours @ 18°C on yeast at cap.

Analysis

- Triangle tests on Reps
- Descriptive sensory analysis on all treatments

Fund Fonds
Fonds Baillet Latour

# SENSORY RESULTS

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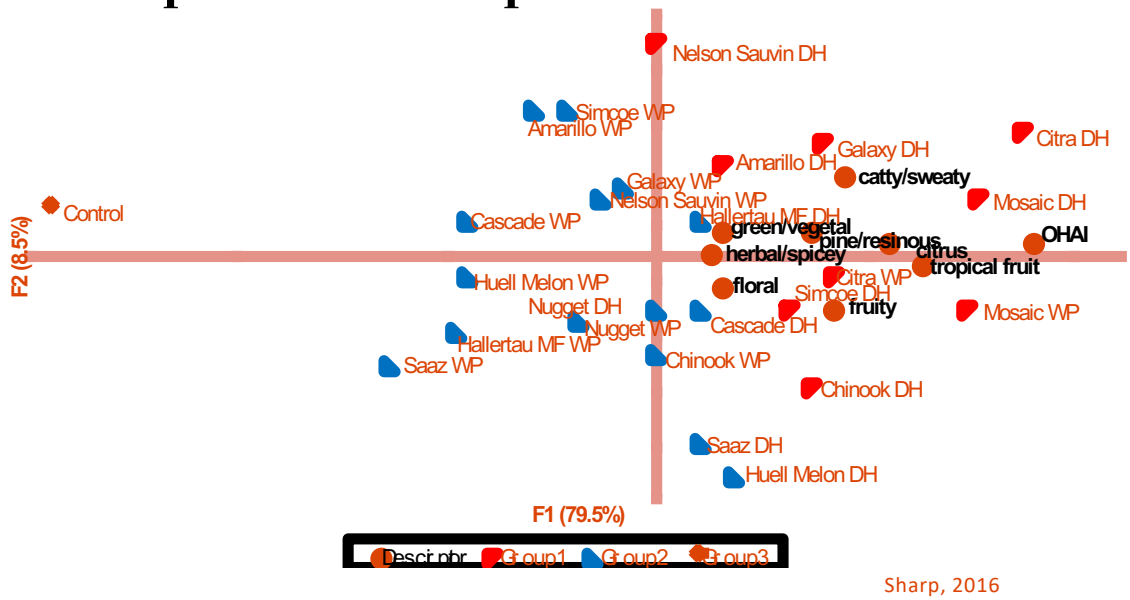
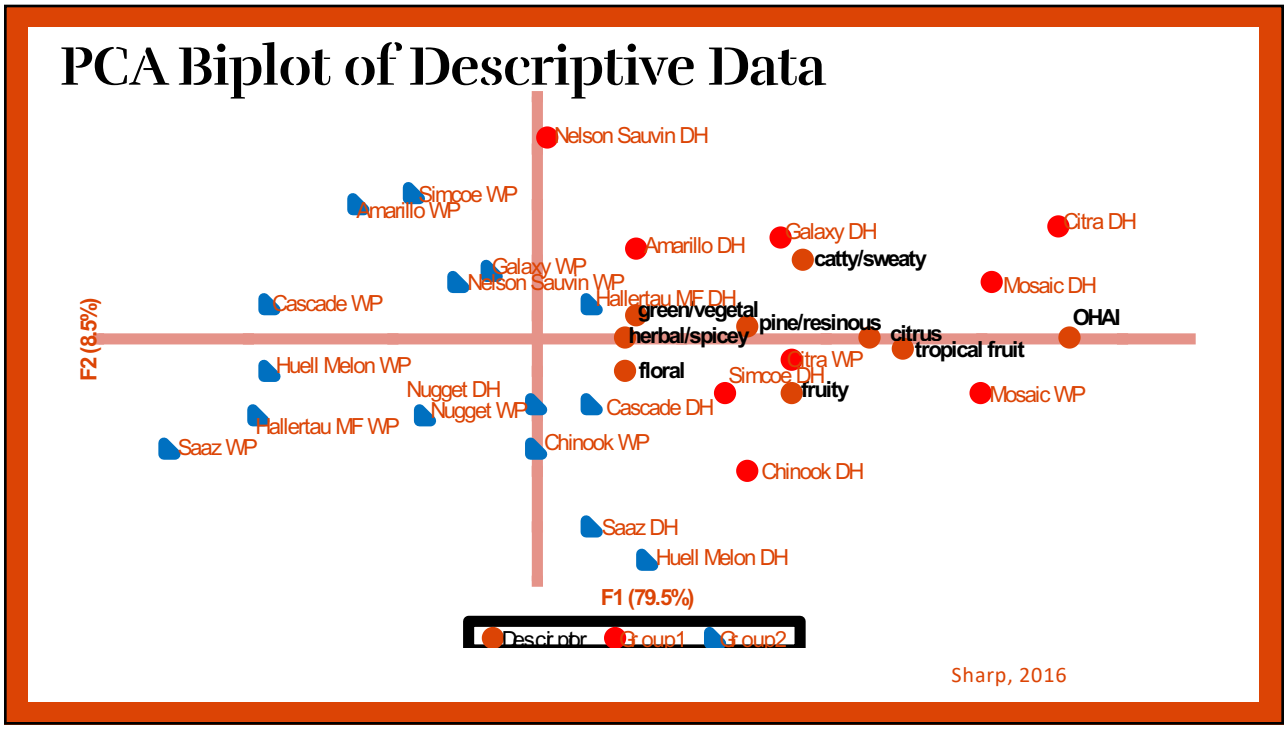
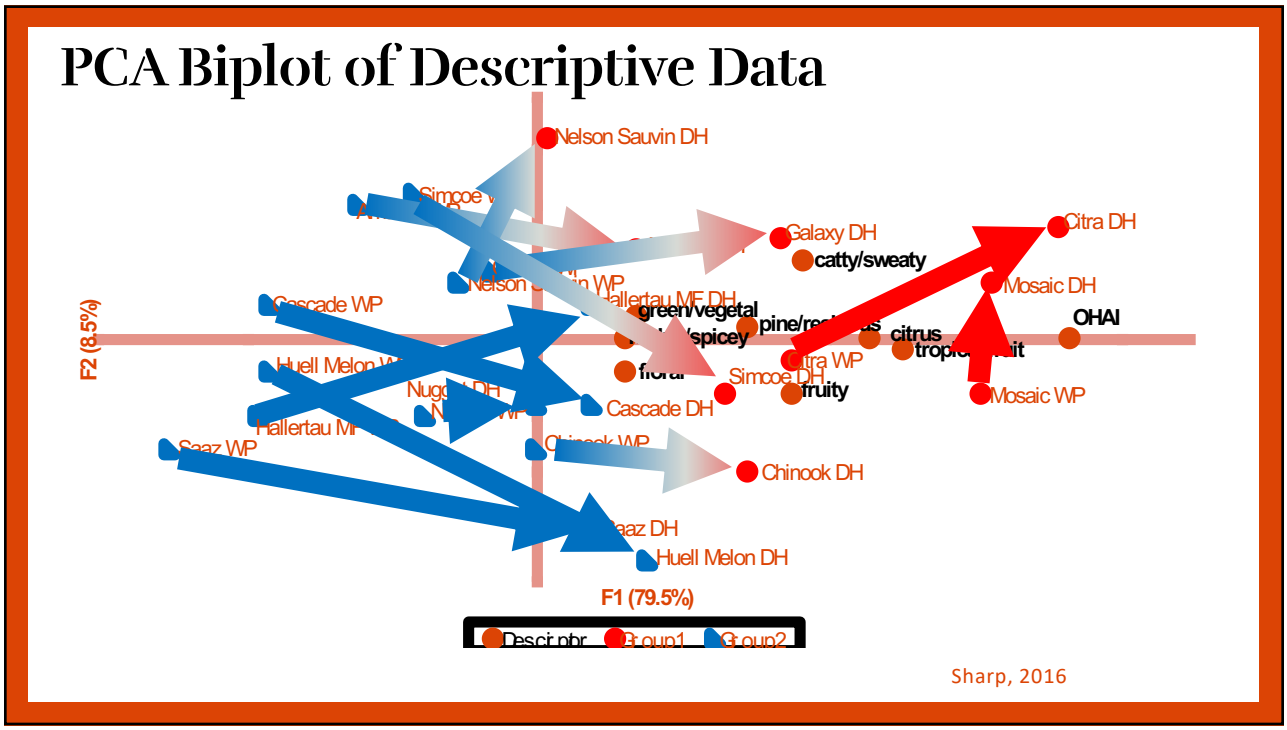
## PCA Biplot of Descriptive Data




PCA Biplot of Descriptive Data
Nelson Sauvin DH
Simcoe WP
Amarillo WP
Galaxy DH
Citra DH
Amarillo DH
catty/sweaty
Galaxy WP
Nelson Sauvin WP
Mosaic DH
Hallertau MF DH
Cascade WP
green/vegetal
pine/resinous
OHAI
herbal/spicey
citrus
tropical fruit
Citra WP
Huell Melon WP
floral
Simcoe DH
fruity
Nugget DH
Mosaic WP
Nugget WP
Cascade DH
Hallertau MF WP
Chinook WP
Saaz WP
Chinook DH
Saaz DH
Huell Melon DH
F2 (8.5%)
F1 (79.5%)
Sharp, 2016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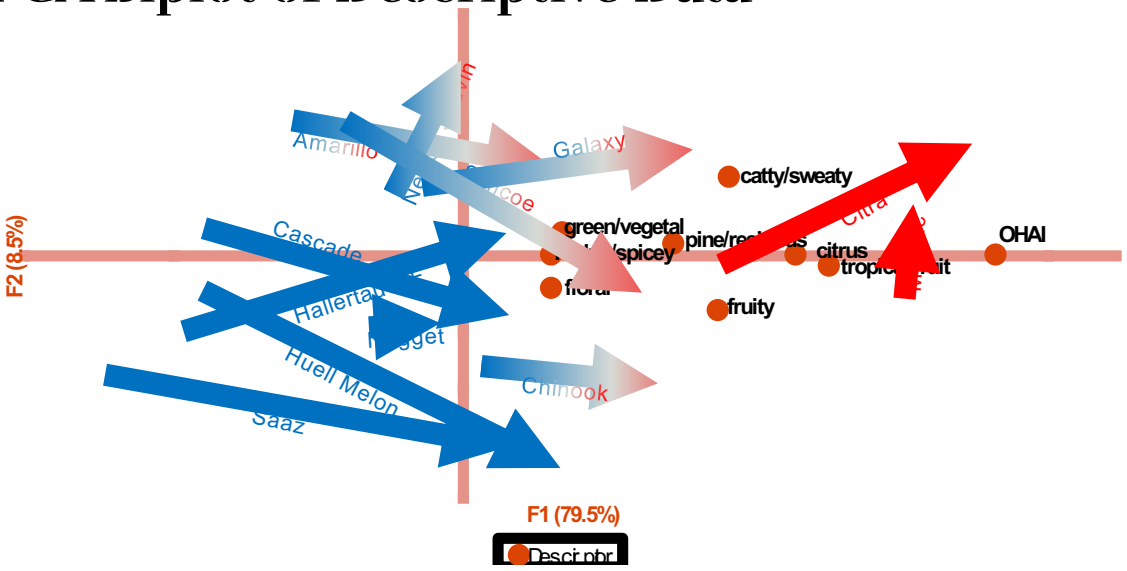
# PCA Biplot of Descriptive Data




Sharp, 2016

# Take-aways

- Extraction efficiencies may be very low
- Dry-hopping increases aromatic intensity relative to late hopping
  - Aroma character may be significantly difference between the two

Scott Lafontaine
Doctoral Candidate
Oregon State University

# DOSING RATES AND EXTRACTION EFFICIENCIES

Oregon State University

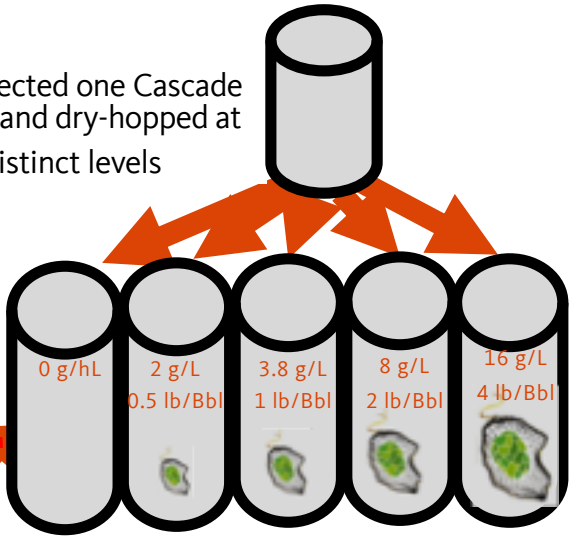
## Background

***Cascade from 2015 Harvest***

**29** *Sample lots*
**19** Farms
**13** Unique oil values
Dry hopped at 3.8g/L

| | Region | Farm (coded) | OSU Hop Oil (ml/100g) |
|---|---|---|---|
| CAS_12_16 | WA | 2 | 0.5 |
| CAS_27_16 | WA | 1 | 0.6 |
| CAS_21_16 | ID | 10 | 0.6 |
| CAS_22_16 | ID | 10 | 0.6 |
| CAS_24_16 | WA | 9 | 0.6 |
| CAS_07_16 | ID | 7 | 0.7 |
| CAS_09_16 | ID | 14 | 0.8 |
| CAS_19_16 | WA | 20 | 0.8 |
| CAS_04_16 | WA | 5 | 0.8 |
| CAS_25_16 | OR | 13 | 0.8 |
| CAS_26_16 | WA | 12 | 0.9 |
| CAS_06_16 | ID | 7 | 0.9 |
| CAS_05_16 | WA | 5 | 1.0 |
| CAS_11_16 | WA | 2 | 1.0 |
| CAS_16_16 | WA | 15 | 1.1 |
| CAS_17_16 | OR | 17 | 1.1 |
| CAS_15_16 | WA | 16 | 1.2 |
| CAS_03_16 | OR | 4 | 1.2 |
| CAS_23_16 | WA | 21 | 1.2 |
| CAS_20_16 | WA | 19 | 1.3 |
| CAS_28_16 | WA | 1 | 1.4 |
| CAS_29_16 | WA | 11 | 1.4 |
| CAS_02_16 | OR | 4 | 1.4 |
| CAS_08_16 | OR | 8 | 1.5 |
| CAS_13_16 | WA | 2 | 1.5 |
| CAS_10_16 | WA | 2 | 1.5 |
| CAS_01_16 | OR | 4 | 1.7 |
| CAS_18_16 | WA | 18 | 1.7 |
| CAS_14_16 | WA | 2 | 2.6 |

Selected one Cascade lot and dry-hopped at 4 distinct levels



## Main objective

The main goal of this project was to determine a dose response curve for Cascade hop aroma.

Does more hop material = more aroma?

## Things to consider when dry-hopping on small scale..

- Sample inhomogeneity
- Dissolved oxygen uptake
- Package scalping

MBAA TQ vol. 53, no. 3 • 2016 • pp. 140–144

PEER-REVIEWED PAPER

**Dry Hopping on a Small Scale: Considerations for Achieving Reproducibility**

**Daniel M. Vollmer and Thomas H. Shellhammer**
Department of Food Science and Technology, Oregon State University, Corvallis, OR 97331, U.S.A.

## Hop Preparation and Dry-Hopping Parameters

- Blend brewer's cuts of whole cone hops by grinding


## Brewing "unhopped" beer

**Beer Specifications:**

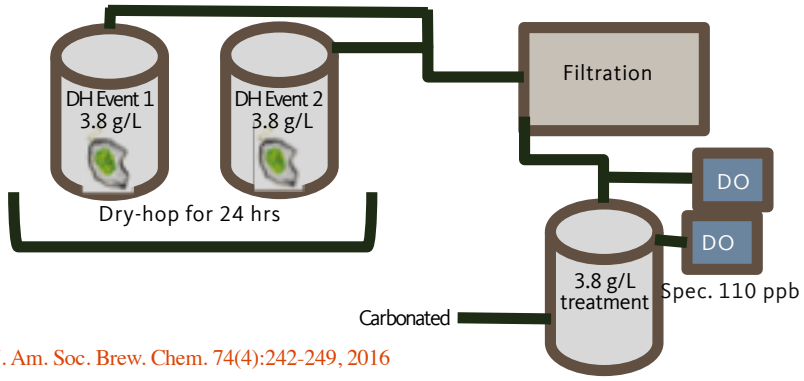
- Grist:
  - 85% Pale 2-row
  - 13.5% Caramel 10L
  - 0.5% Caramel 120L
- **Original Gravity:** 10.6 P
- **Real Extract:** 3.16 P
- **BU** = 20 mg/L (iso-extract)
- **ABV** = 4.8 % ABV

WIDMER BROTHERS BREWING

BRIDGEPORT BREW PUB Portland OREGON

## OSU's current small-scale dry-hopping process

- All dry-hop events occur in duplicate (40 L beer each)
- During filtration 2 kegs are blended during filtration into 1 keg
  - Oxygen monitoring




Vollmer, D. et. al. J. Am. Soc. Brew. Chem. 74(4):242-249, 2016

## Evaluations using draft beer

- Minimized total package oxygen
- Great for sensory testing implementation

# Sensory evaluation - descriptive analysis

# Sensory evaluation – descriptive analysis external controls

| | *Attributes* | Base (No dry hop) | 3.8 g/L | 16 g/L | Ballast Point Grapefruit Sculpin | Hop Valley Citrus Mistress |
|---|---|---|---|---|---|---|
| Assess Descriptors Based on Aroma Only | Overall Hop Aroma Intensity | 0 | 8-9 | 14-15 | 14-15 | 7-8 |
| | Citrus | 0 | 7-8 | 5-6 | 13-14 | 6-7 |
| | Herbal/Tea | 0 | 5-6 | 12-13 | 1-2 | 6-7 |

- **Panelists came to consensus for attributes on commercial and internally made samples**
- **References were served to panelists at each DA session**

# SENSORY RESULTS

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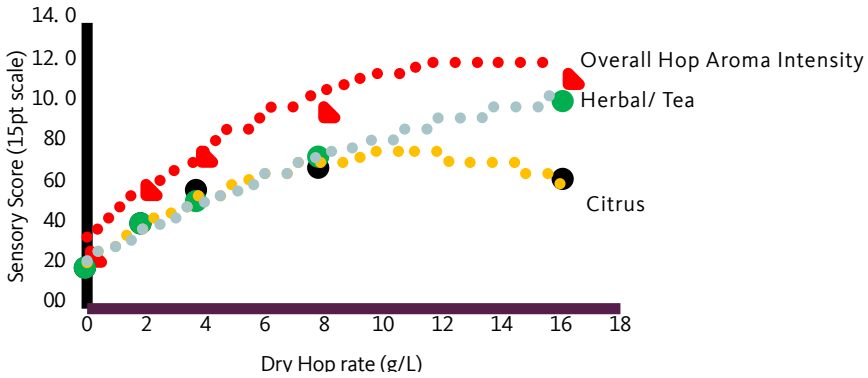
## Hop dose response – hoppy quality (citrus and herbal/tea)



# CHEMISTRY RESULTS

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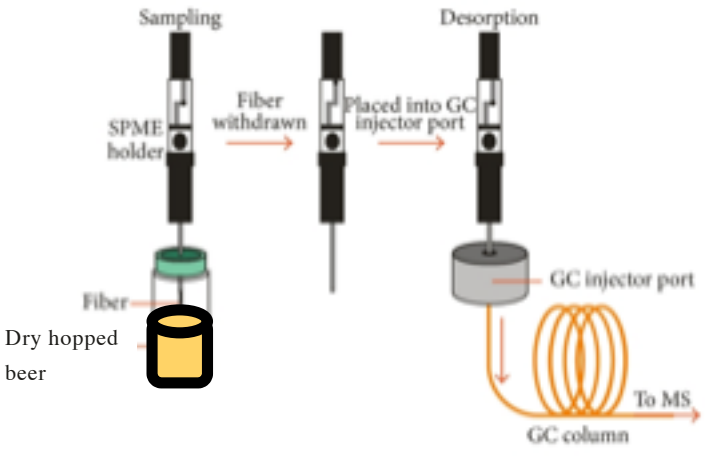
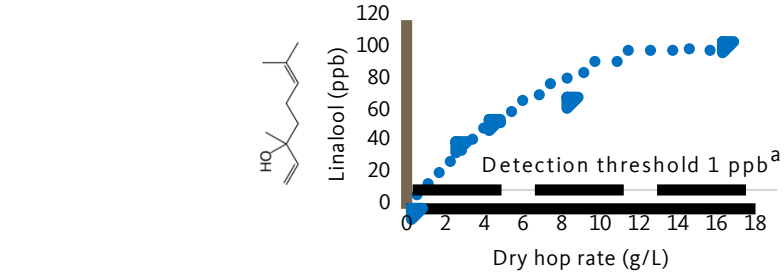
## Solid phase micro extraction GC/MS – Hop volatiles (In Beer)

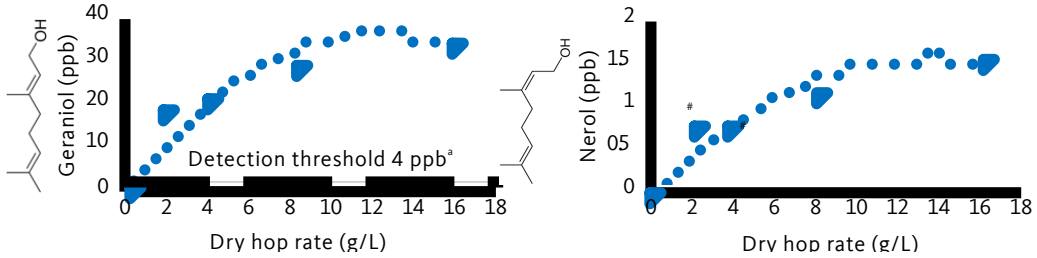



| Internal Standard |
|---|
| 4-octanol |
| **Target Hop Analytes** |
| Linalool |
| Terpinen-4-ol |
| α-terpineol |
| Nerol |
| Phenyl Acetate |
| Geraniol |
| Geranial-citral |
| Methyl Geranate |
| Geraniol Acetate |
| β-Caryophellyene |
| α-Humulene |
| β-Farnesene |
| Gernyal Isobutyrate |

# Hop Compound Concentrations – Terpene Alcohols



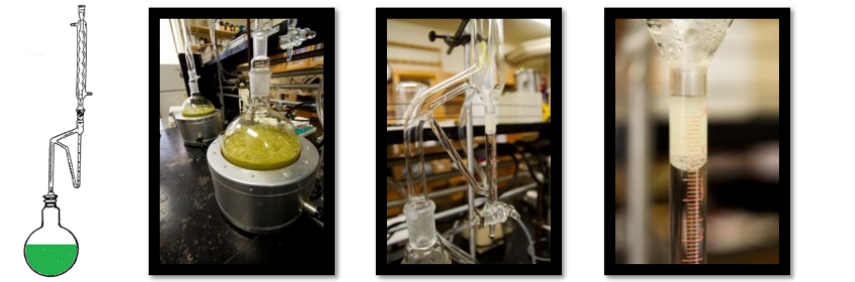

[a]Kishimoto J. Agric. Food Chem Vol 54, no 23. 2006




*Average of 4 SPME-GC/MS instrumental runs

#Estimated values lower than LOQ

# Hop total oil + compositional analysis



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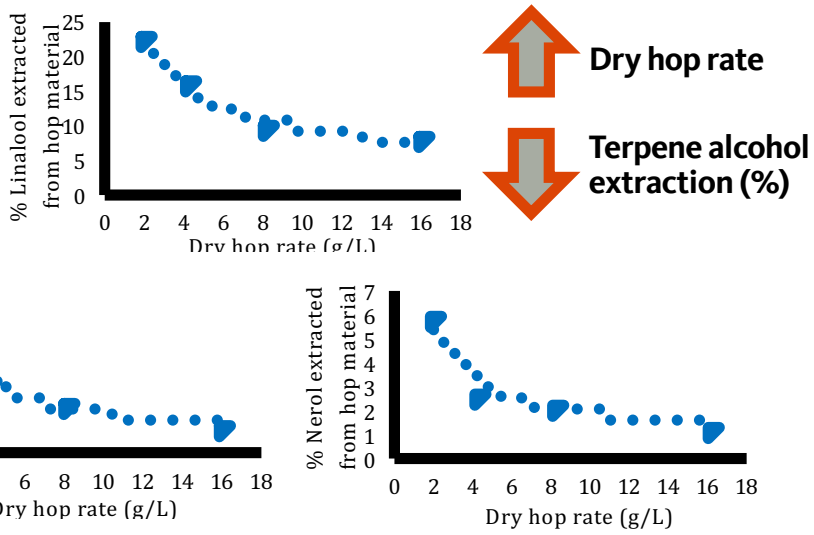
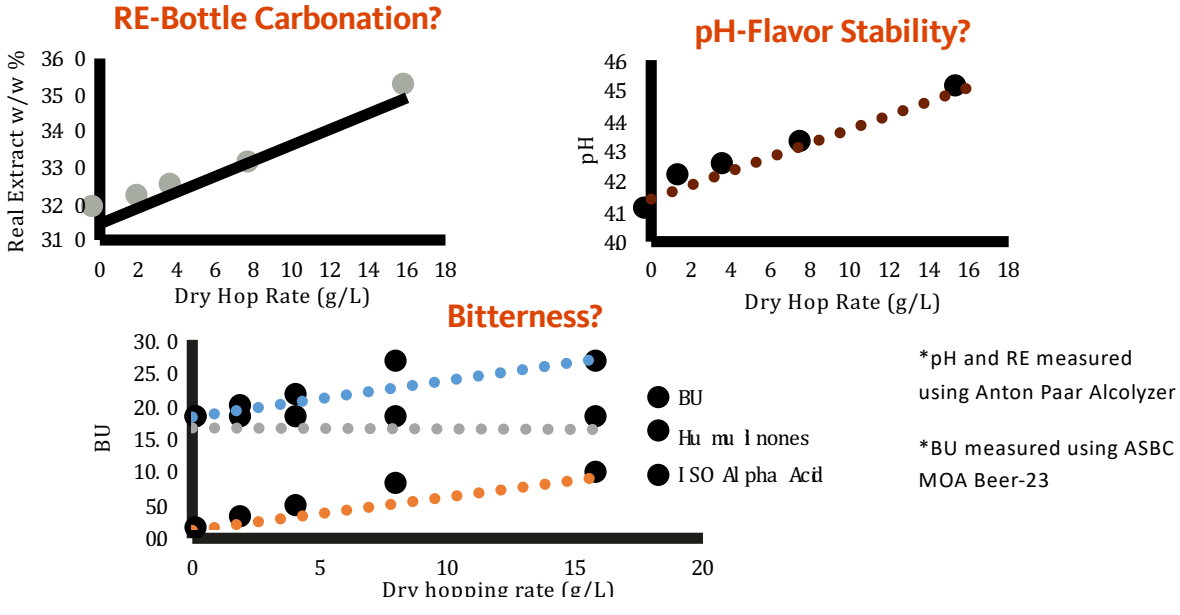
# Hop Compound % Extraction – Terpene Alcohols




*Assuming 100% extraction based on hop oil hydrodistillation

# Dry hopping having other impacts on beer.....




*pH and RE measured using Anton Paar Alcolyzer

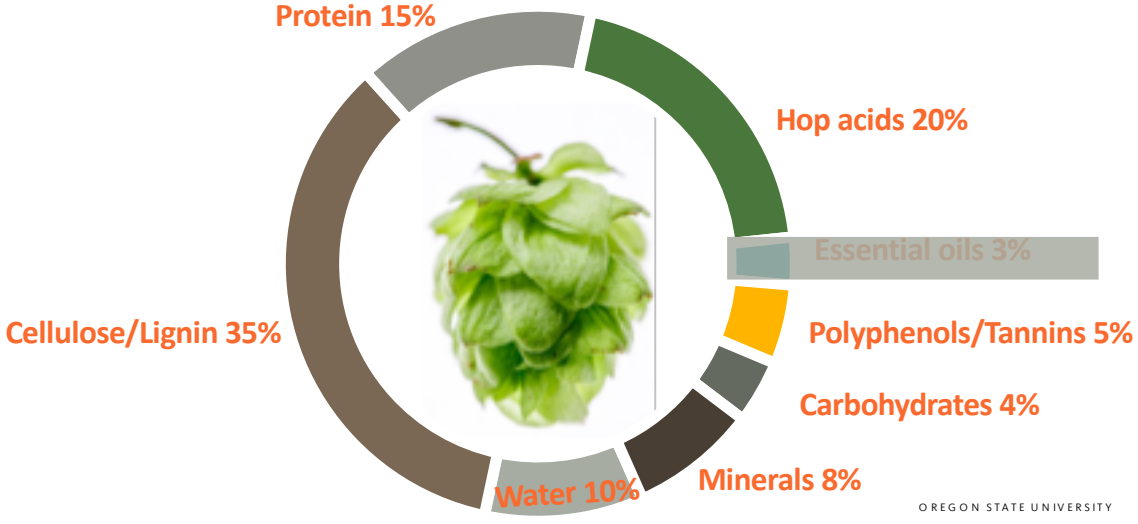
*BU measured using ASBC MOA Beer-23

Thomas H. Shellhammer, PhD
Nor'Wester Professor of Fermentation Science
Oregon State University

# BITTERNESS OF DRY-HOPPED BEER

Oregon State University

## How does the nonvolatile fraction influence dry-hopped beer quality?

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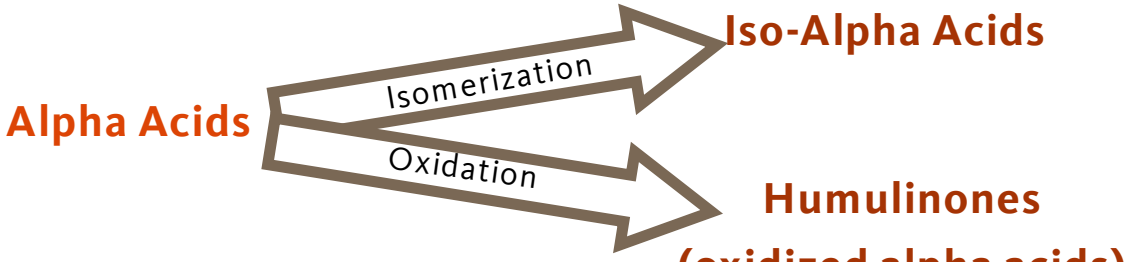
## Hop acids (up to ~20% of hops)

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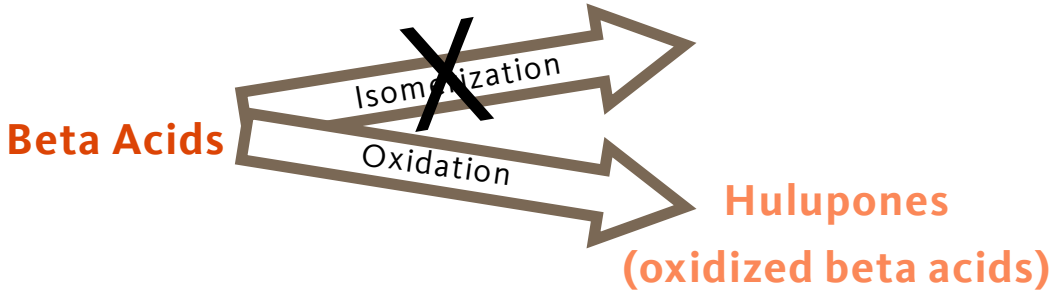


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# Hop acids – what you may find in beer

**Iso-Alpha Acids**

**Alpha Acids**

**Humulinones (oxidized alpha acids)**

~~Hulu~~ones (oxidized beta acids)

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# Regarding dry-hopped beers… Does BU work? What drives bitterness?

**Beer**: 121 unique brands from 42 breweries

- 30 brands multi rep study + 91 brands single rep study

**Chemical analysis**: 7 factors

- Iso-alpha acids, oxidized hop acids, alpha acids, TPP,
- ABV, RE, pH
- BU

**Sensory analysis**:

- Bitterness intensity
- Multiple Replication study: data for model building
- Partial Replication study: data for model testing

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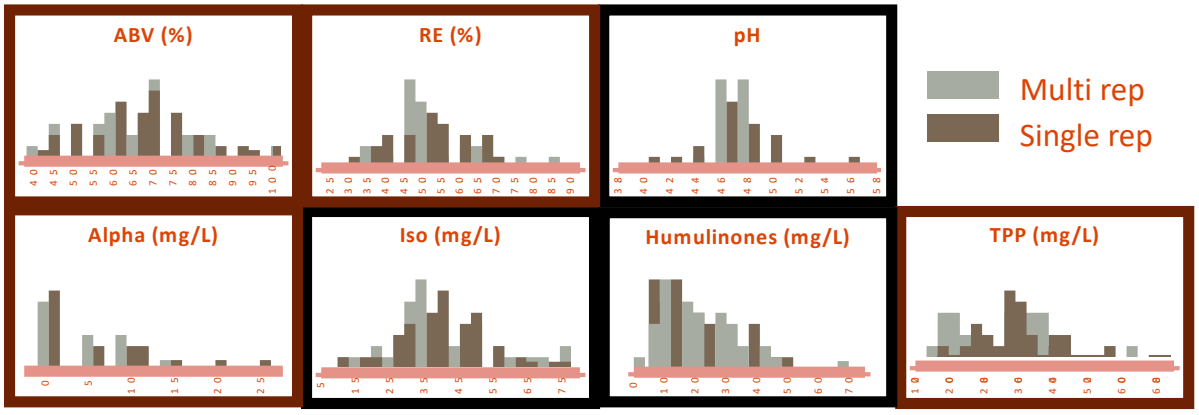
# Chemistry of beers in commercial survey

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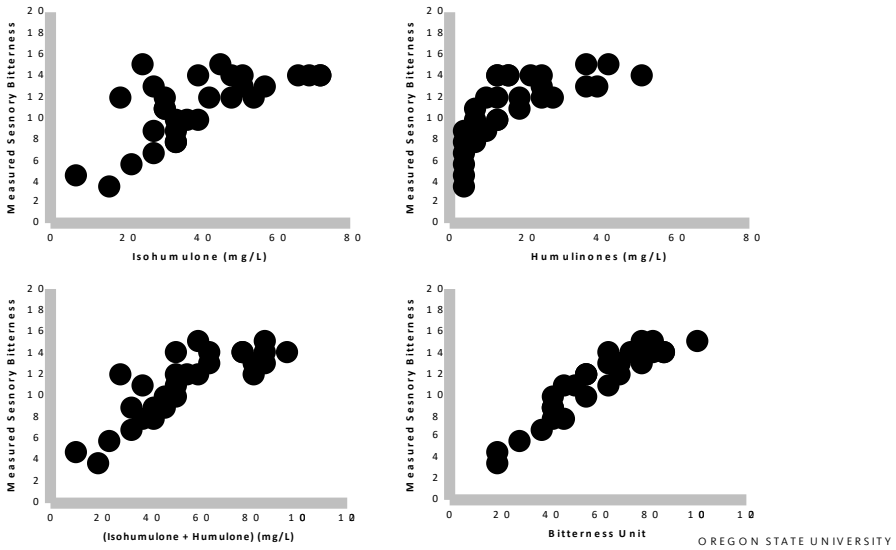
# Bitterness comes from Isos & Humulinones
# BU predicts bitterness

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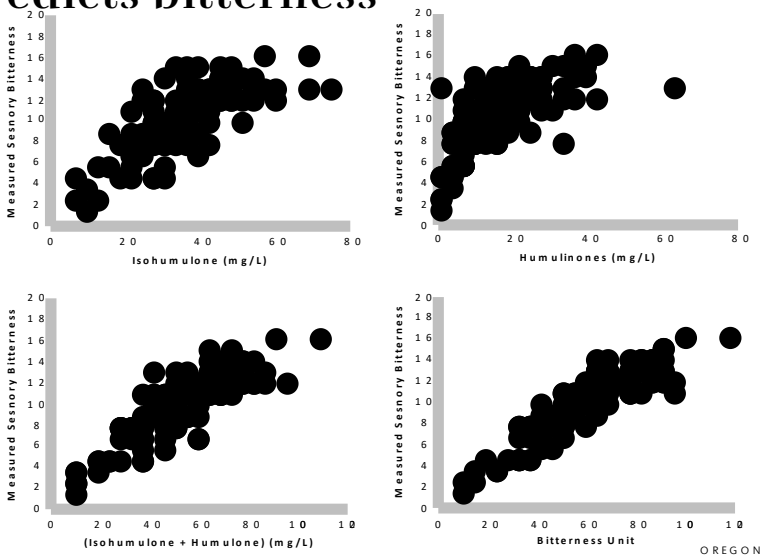


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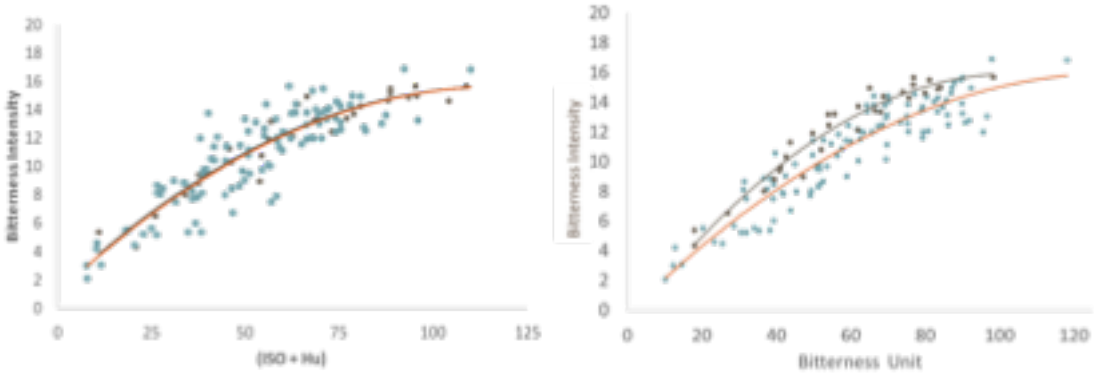


Hahn, C, Lafontaine, S.R., Pereira, C.B. and Shellhammer, T.H. 2018. Evaluation of the Nonvolatile Chemistry Affecting the Sensory Bitterness Intensity of Highly Hopped Beer, Journal of Agricultural and Food Chemistry

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# HOP CREEP

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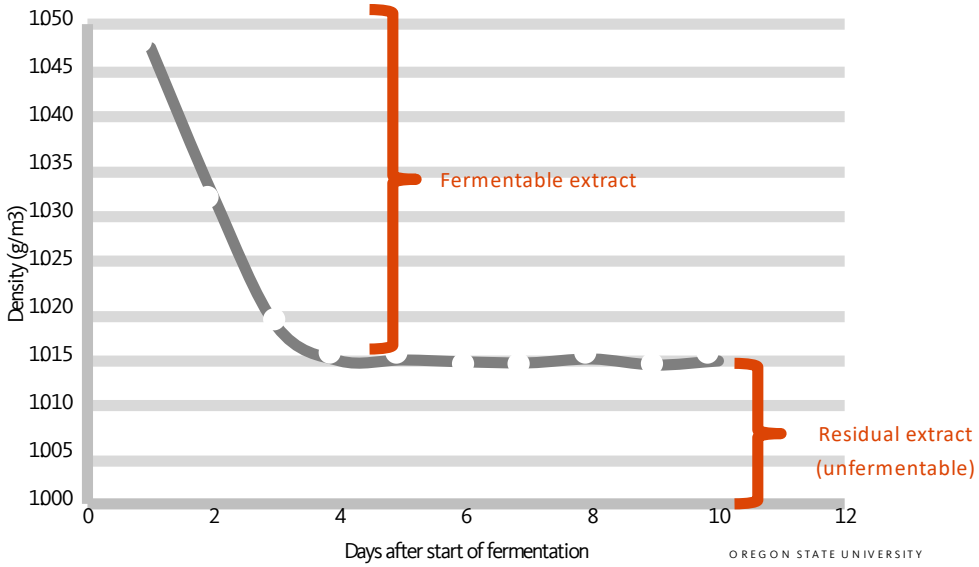
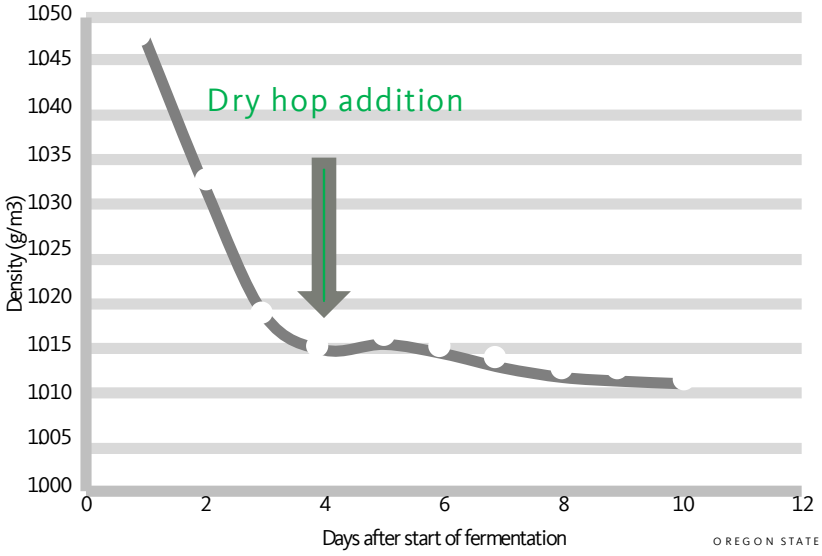
## Typical fermentation, no dry-hopping



# Dry-hopping can create "Hop Creep"

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# Cascade hops have broad (low) enzyme activities

| Enzyme | Hops | Malt (130 dp) |
|---|---|---|
| α-amylase | 0.35 | 198 |
| β-amylase | 0.41 | 13 |
| Amyloglucosidase | 0.02 | NA |
| Limit dextrinase | < 0.01 | NA |

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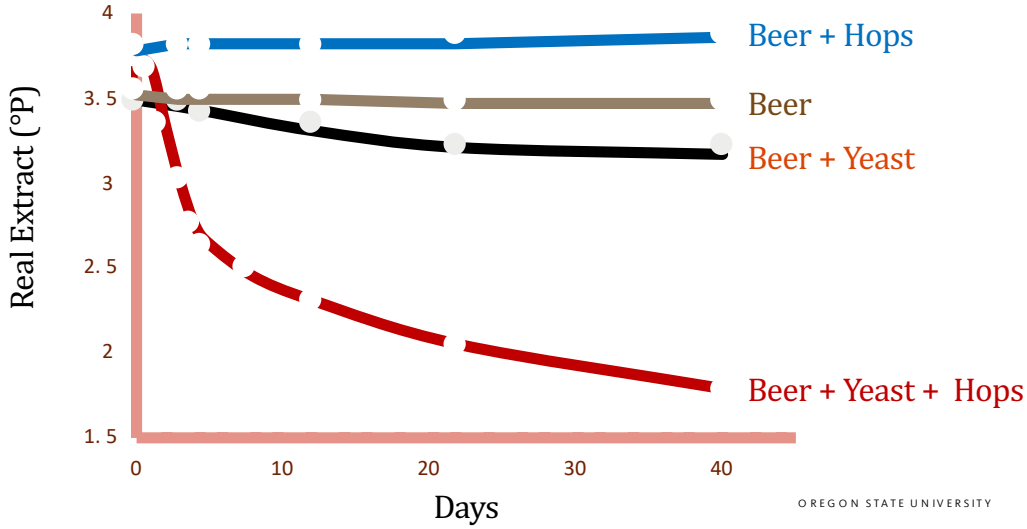
## Hop enzymes stimulate "after-fermentation" AKA – Hop Creep

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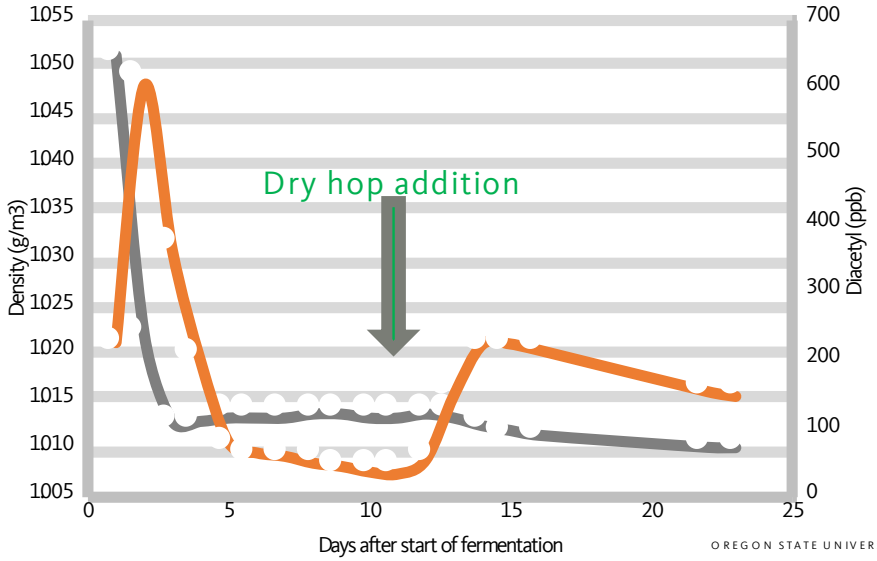
## Hop Creep & Diacetyl issues

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# HOP ENZYMES PERSIST IN PACKAGED BEER

Oregon State University

## Enzyme action during production and post-packaging

**Dry hopping schedule:**

- **2** days after yeast harvest
- Dry hop warm
- **2-4 lb/bbl** hops
- **2** dry hop additions
- **7** days on hops

**Finishing:**

- Crash cool
- Centrifuge
- Up to 24 hours hold prior to packaging

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# Sampling plan

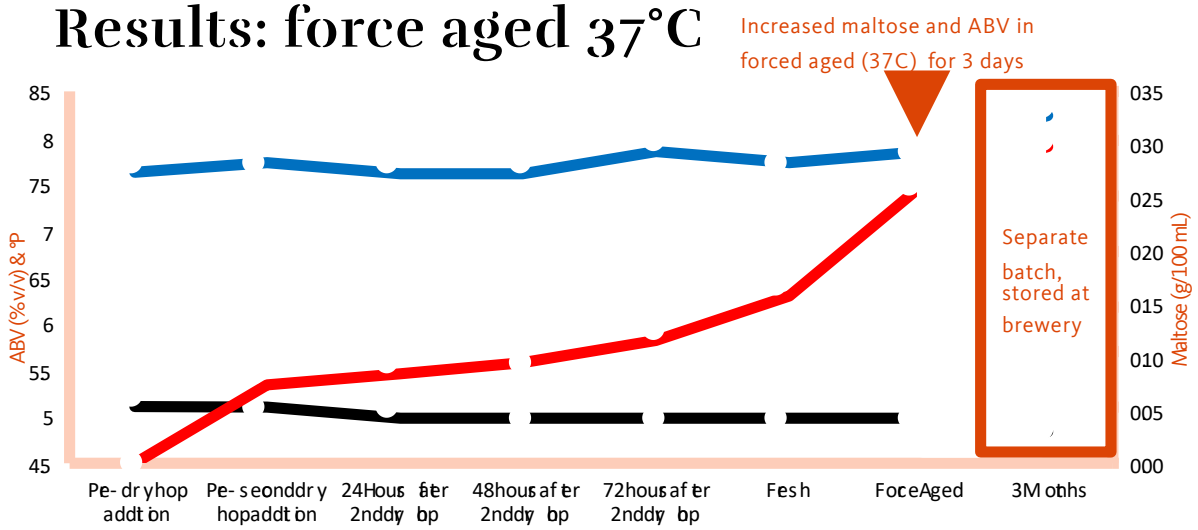
Samples measured on Anton Paar Alcolyzer/Densitometer & HPLC

In-process samples:
- Pre-dry hop addition
- Pre-second dry hop addition
- 24 hours
- 48 hours
- 72 hours

Finished beer samples:
- Fresh
- Force aged (3 days @ 37° C)
- 3 months 25C (packaged 3 months earlier)

ABV (% v/v) Maltose (g/100 mL) RE (°P)

# Results: force aged 37°C

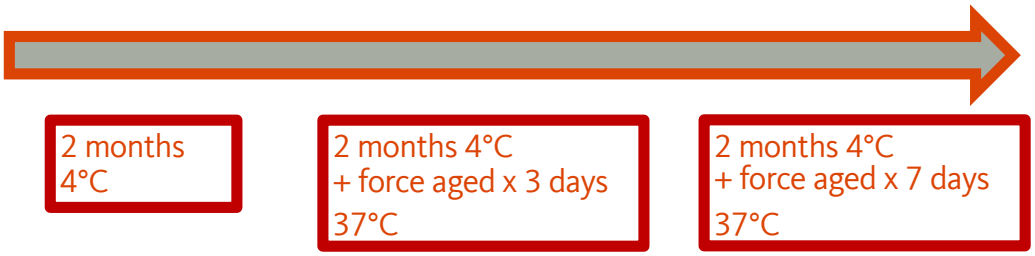



ABV (% v/v) Maltose (g/100 mL) RE (°P)

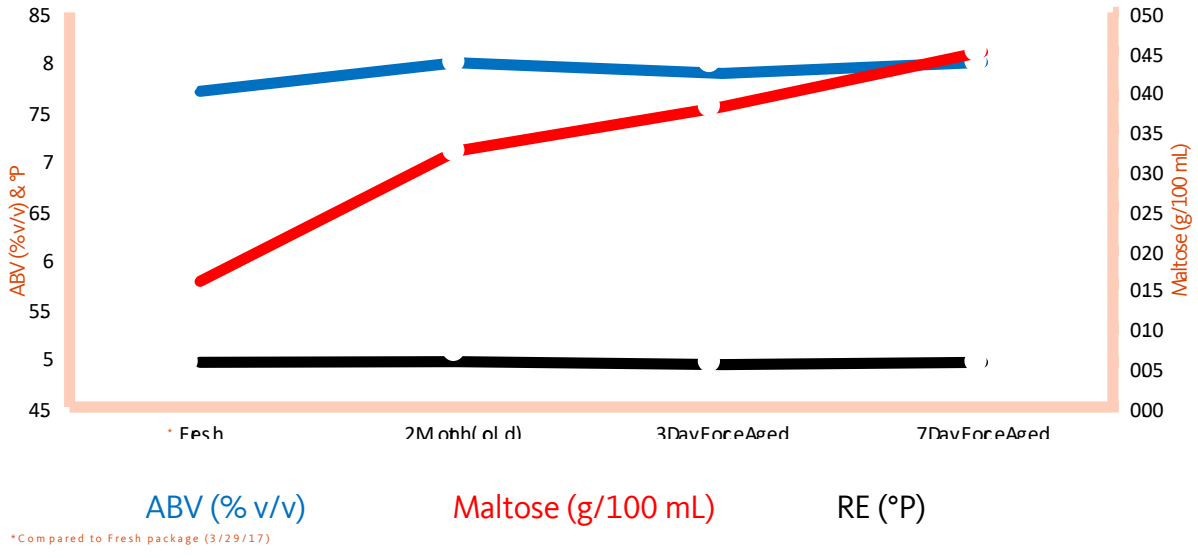
# Continued aging study – 2 months later

- Same packages tested 2 months later
- Force aging repeated (at 37°C) for 3 and 7 days

**Do enzymes transferred into beer after dry-hopping and continue to reduce beer limit dextrins?**




# Repeat force aging study – 2 months later




*Compared to Fresh package (3/29/17)

# Conclusion

- Humulinones coming from hops (during processing and storage) can significantly impact dry-hopped beer bitterness
- Hop-derived enzymes can alter carbohydrate make up of Real Extract
  - Refermentation in the presence of yeast (for example – bottle conditioning)
  - Lead to diacetyl spikes
  - SOLUTION: dry hop timing, temperature, hop variety, pasteurization
- Hop enzymes persist in finished beer
  - Dry-hopped beers likely become sweeter with age

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# Acknowledgements

**Oregon State University**
Jeff Clawson
Kaylyn Kirkpatrick
Andrew Sutton
Cameron McDaniel
Dan Vollmer

**Hops**
John I Haas
Yakima Chief HopUnion
Crosby Hop Farm

**Funding agencies**
Fonds Baillet Latour Fund
Hop Research Council

**Breweries**
Allagash Brewing Company
Craft Brew Alliance
Bridgeport Brewery
Ninkasi Brewing Company
Russian River Brewing Company
pFriem Brewing Company
Melvin Brewing Company

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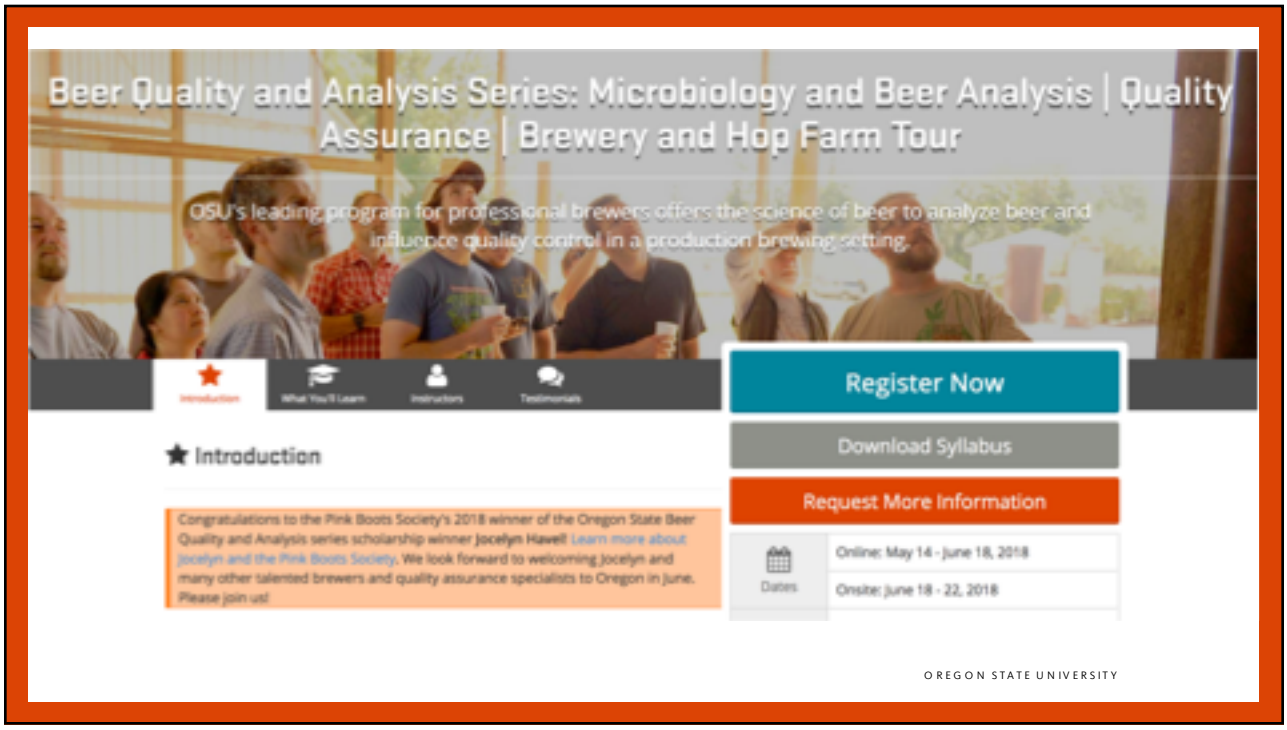


Thank you

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