Dry-hopping Beer and Achieving Consistent Flavor

Shellhammer Laboratory
Department of Food Science and Technology
Oregon State University

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Director of Brewing Operations
Ninkasi Brewing Company

WHIRLPOOL VS. DRY- HOPPING: SENSORY ANALYSIS

Hop Aroma:
Late vs. Dry Hopping

- Background
- Basics of Experimental Design and Setup
- Results

Background and literature review

J. Inst. Brew., March–April, 1983, Vol. 89, *pp.* 87–91

DIFFERENCES IN UTILISATION OF THE ESSENTIAL OIL OF HOPS DURING THE PRODUCTION OF DRY-HOPPED AND LATE-HOPPED BEERS

BY J. HALEY* AND T. L. PEPPARD

(*Brewing Research Foundation, Lyttel Hall, Nutfield, Surrey*)

Background and literature review

Hop Aroma Component Profile and the Aroma Unit

Gail B. Nickerson, *Department of Agricultural Chemistry, Oregon State University, Corvallis 97331-6502,* **and Earl L. Van Engel,** *Blitz-Weinhard Brewing Company, Portland, OR 92709*

Background and literature review

Effect of Hopping on the Headspace Volatile Composition of Beer[1]

Aki A. Murakami, Sydney Rader, Etzer Chicoye, and Henry Goldstein, *Miller Brewing Company, Technical Center, 3939 West Highland Blvd., Milwaukee, WI 53201*

Background and literature review

JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

J. Agric. Food Chem. **2006,** *54,* 8855–8861

Comparison of the Odor-Active Compounds in Unhopped Beer and Beers Hopped with Different Hop Varieties

TORU KISHIMOTO,* AKIRA WANIKAWA, KATSUNORI KONO, AND KAZUNORI SHIBATA

Background and literature review

BrewingScience November / December 2016 (Vol. 69)

Sharp, D. C., Qian, Y., Clawson, J. and Shellhammer, T. H.

An exploratory study toward describing hop aroma in beer made with American and European Hop Cultivars

Background and literature review

MBAA TQ vol. 54, no. 1 • 2017 • pp. 2–10

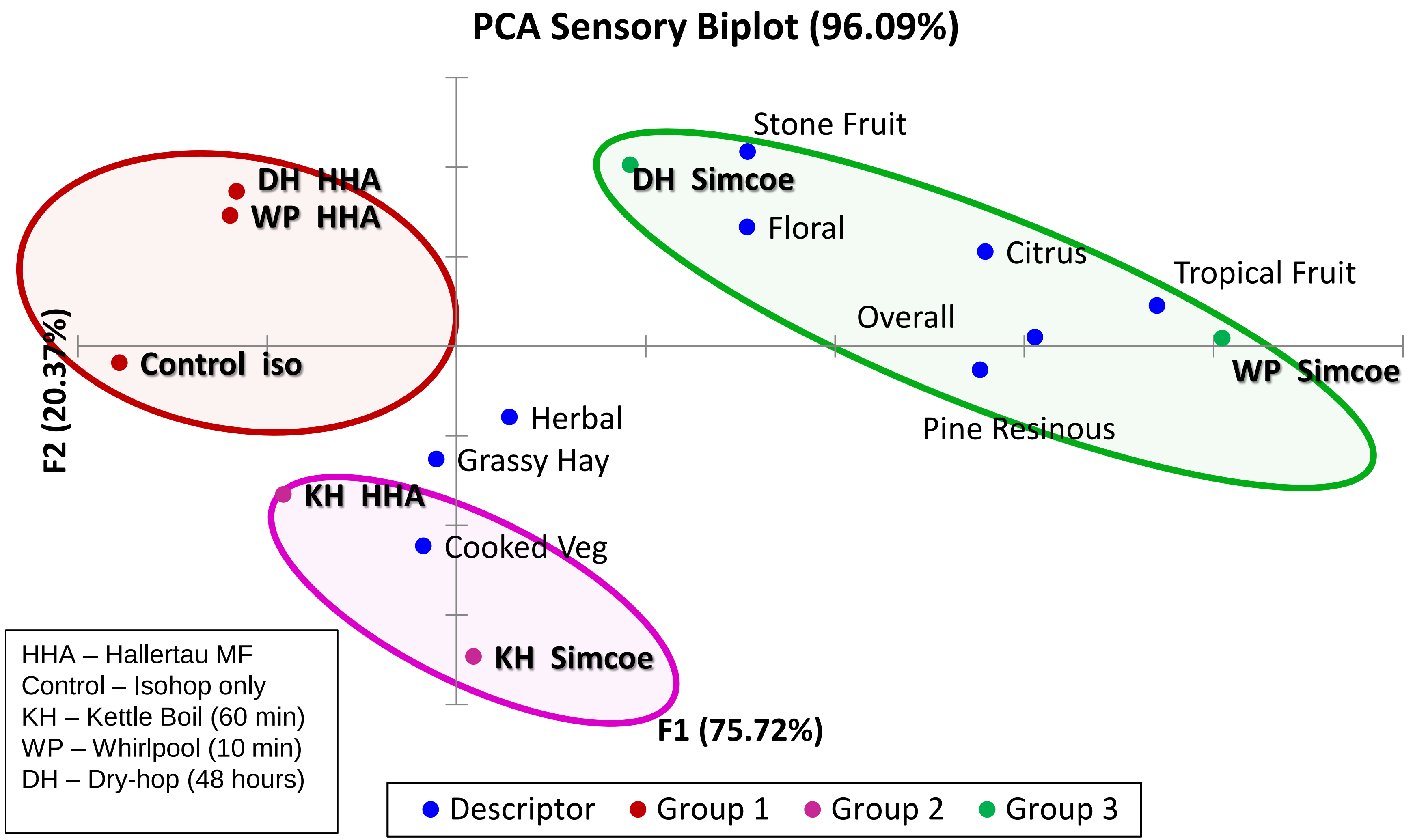
PEER-REVIEWED PAPER

Comparison of the Contributions of Hop Pellets, Supercritical Fluid Hop Extracts, and Extracted Hop Material to the Hop Aroma and Terpenoid Content of Kettle-Hopped Lager Beers

Daniel C. Sharp,[1] YanPing Qian,[2] Jeff Clawson,[1] and Thomas H. Shellhammer[1]

1. Oregon State University Department of Food Science and Technology, 100 Wiegand Hall, Corvallis, OR, U.S.A.
2. Oregon State University, Crop and Soil Sciences, 455 Crop and Soil Sciences, Corvallis, OR, U.S.A.

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: 10.1094/ASBCJ-2017-2144-01

EXPERIMENTAL DESIGN

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 = 50 units *3 Kettle boils (* 6 sensory reps = 300 samples.
- Brew length

Experimental Design

Experimental Controls

- Common wort stream
- Psuedo Replication
- Split Kettle Boils
    - Blocked treatments
    - Small Treatment vessels

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.

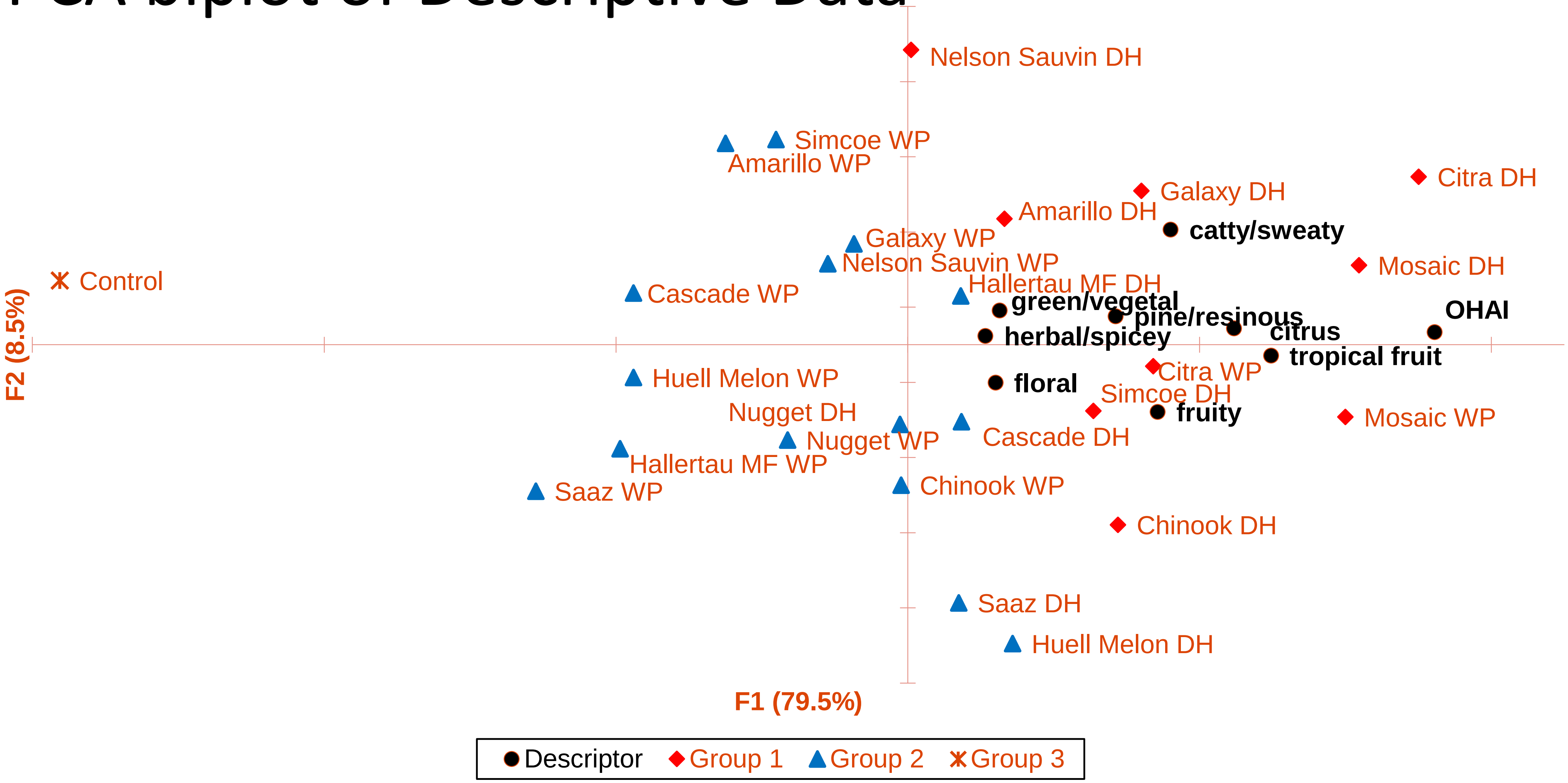
Analysis

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

Fund Fonds
Fonds Baillet Latour

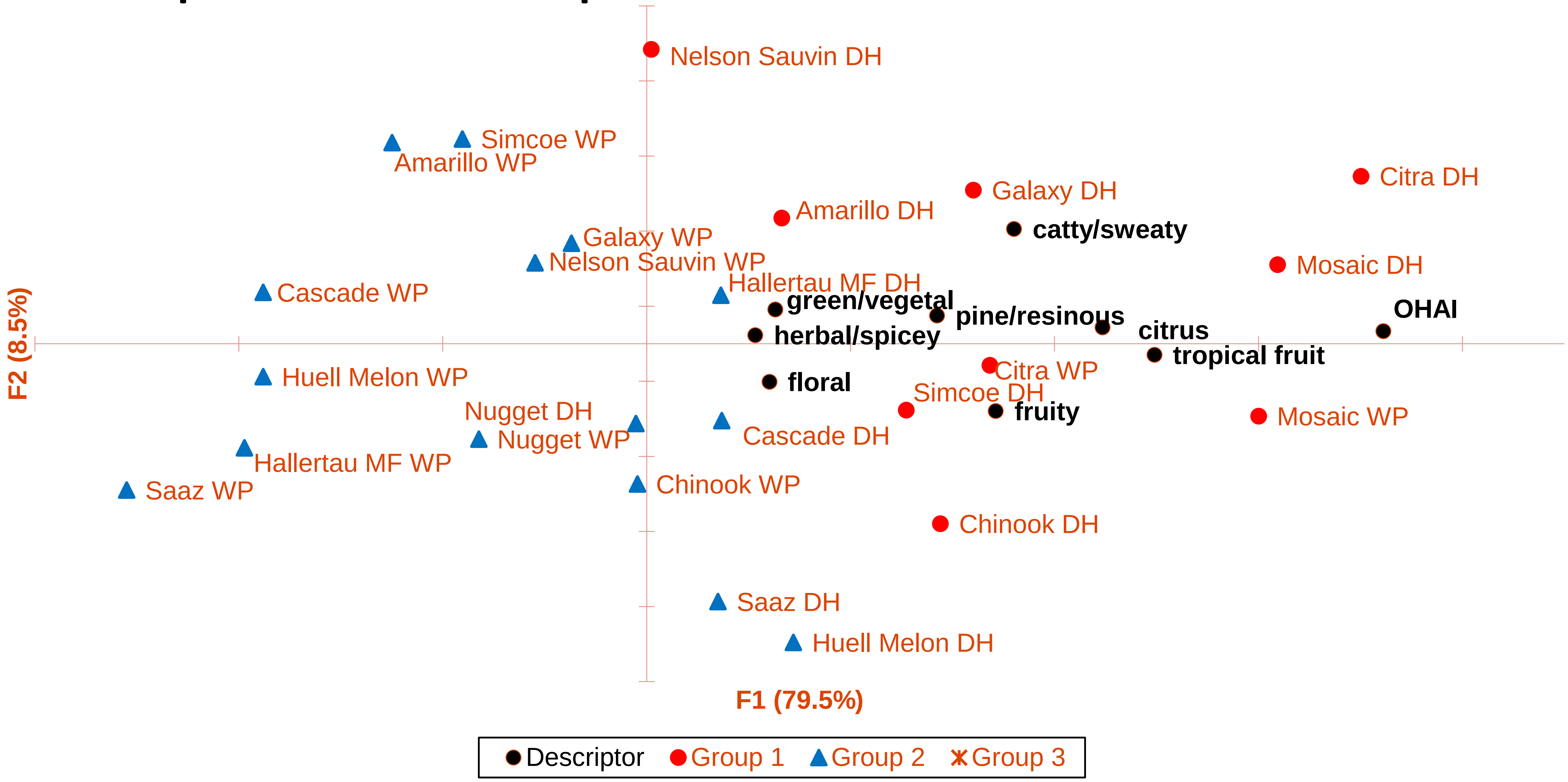
SENSORY RESULTS

PCA biplot of Descriptive Data



Sharp, 2016

PCA biplot of Descriptive Data



Sharp, 2016

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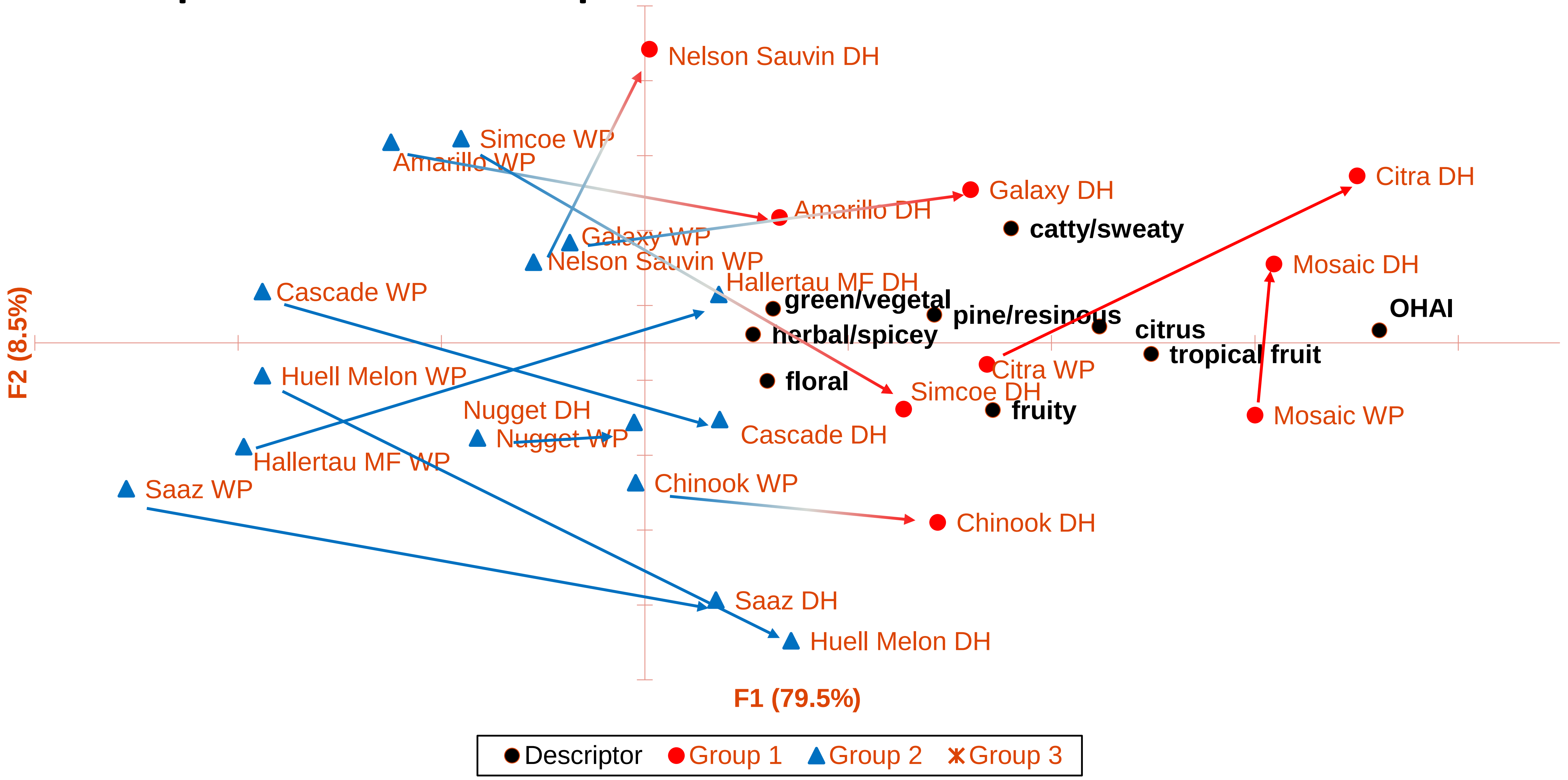

Sharp, 2016

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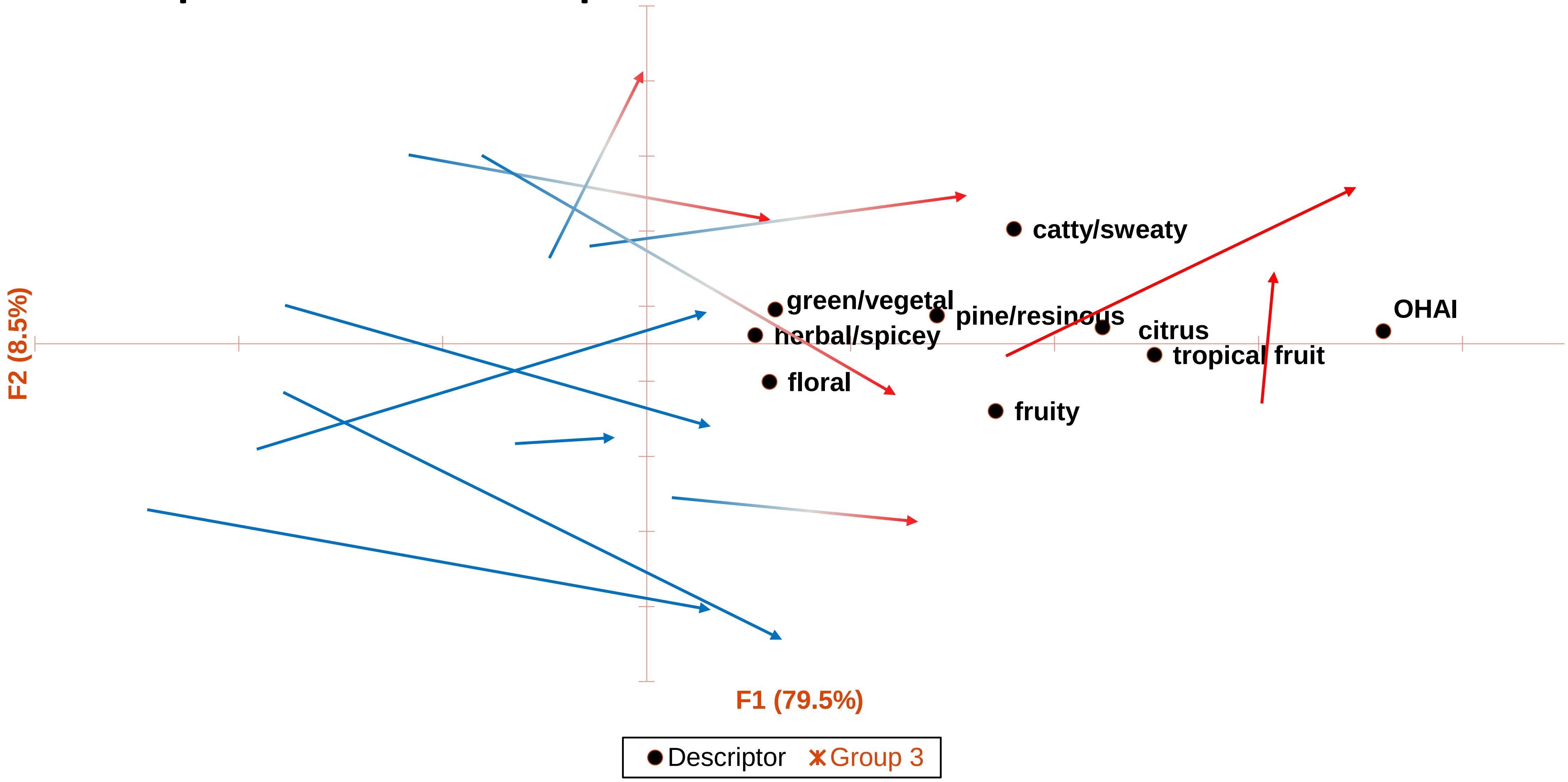

Sharp, 2016

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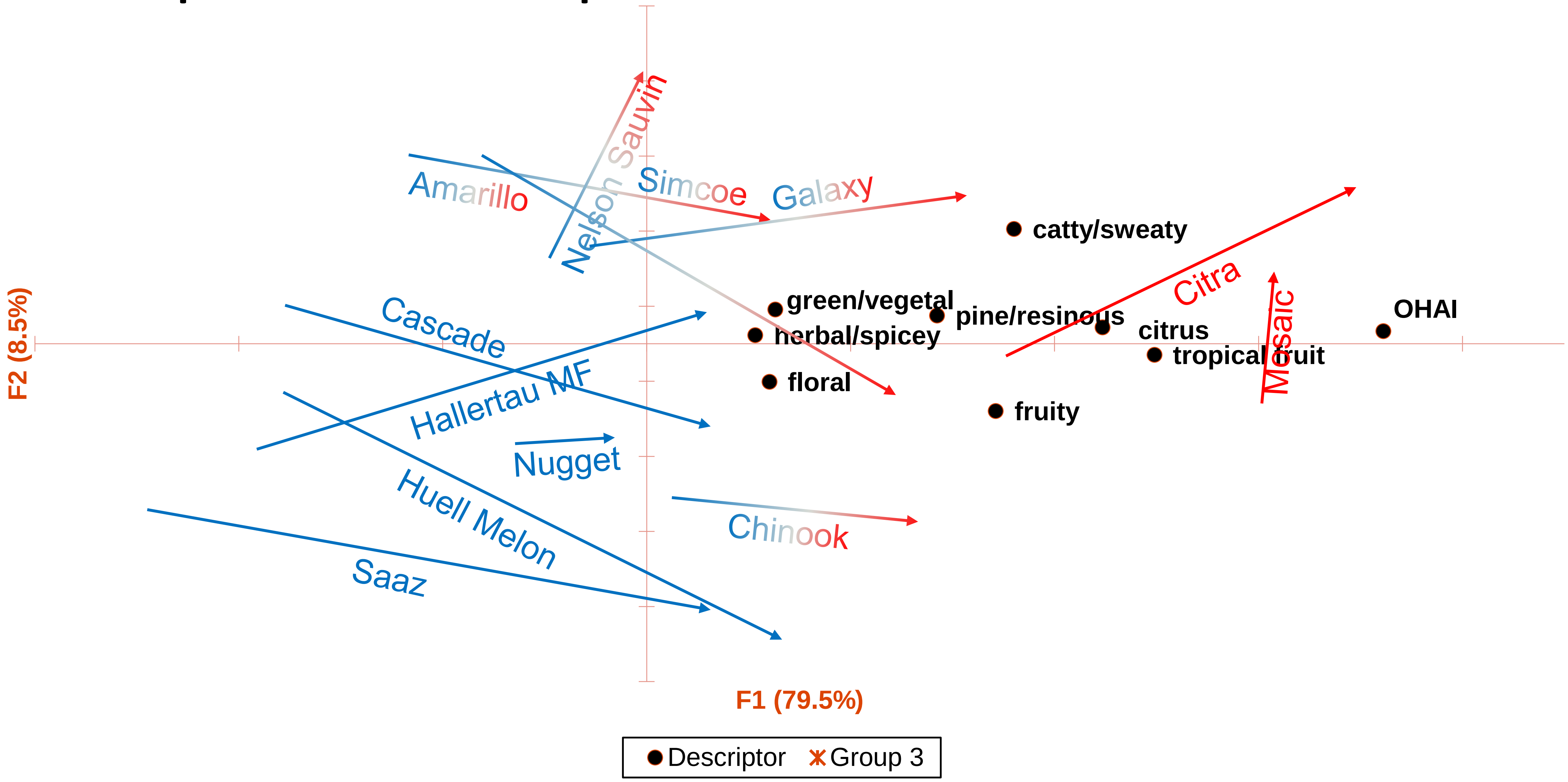

Sharp, 2016

Take-aways

- Normal process variation = minimal effect
- 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

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

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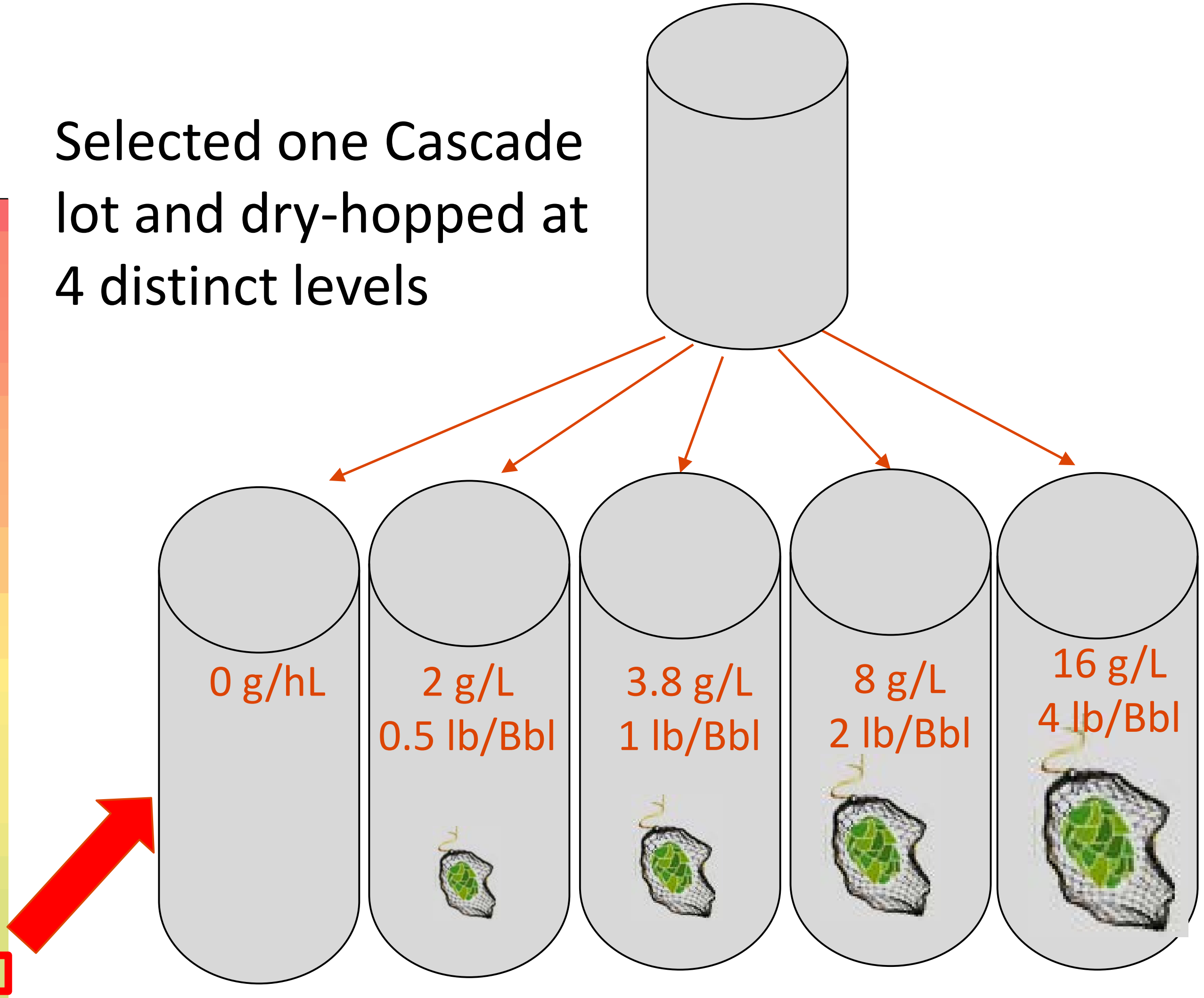
CAS_14_16	WA	2	2.6

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.

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% Carmel 10L
  - 0.5% Carmel 120L
- **Original Gravity:** 10.6 P
- **Real Extract:** 3.16 P
- **BU** = 20 mg/L (iso-extract)
- **ABV** = 4.8 % ABV

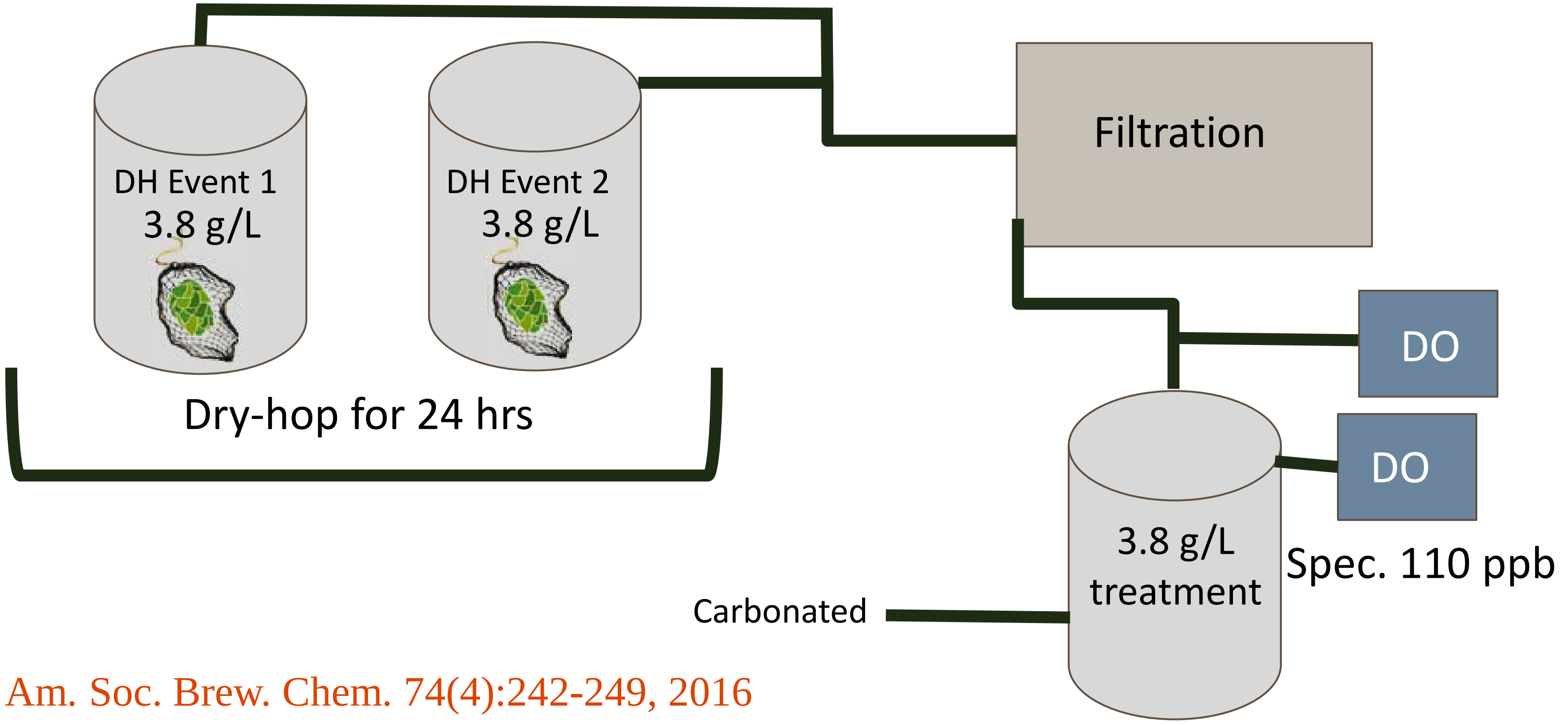
WIDMER BROTHERS BREWING

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

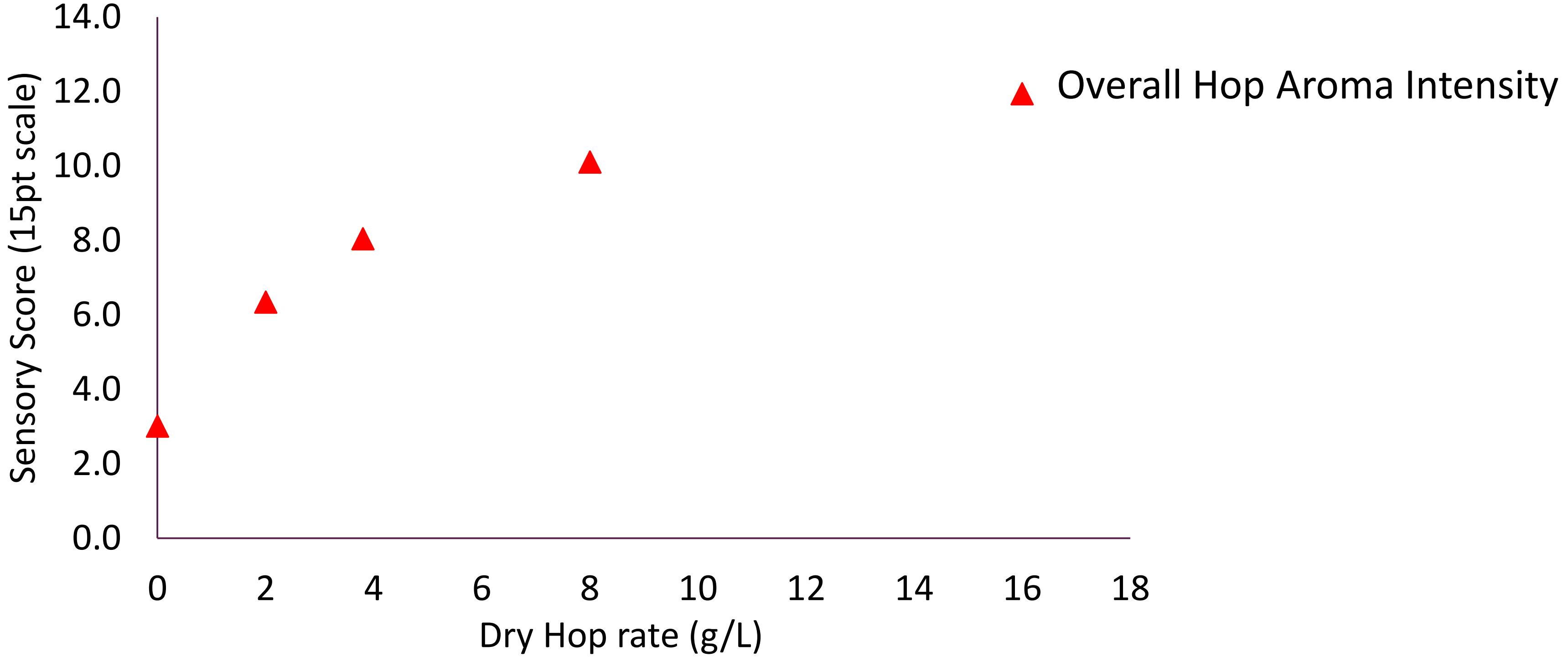
	Attributes	Base (No dry hop)	3.8 g/L	16 g/L	Ballast Point Grapefruit Sculpin	Hop Valley Citrus Mistress
Assess Descriptors Based on Aroma Only	Overall Hop Aroma Intensity	0	8-9	14-15	14-15	7-8
	Citrus	0	7-8	5-6	13-14	6-7
	Herbal/Tea	0	5-6	12-13	1-2	6-7

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

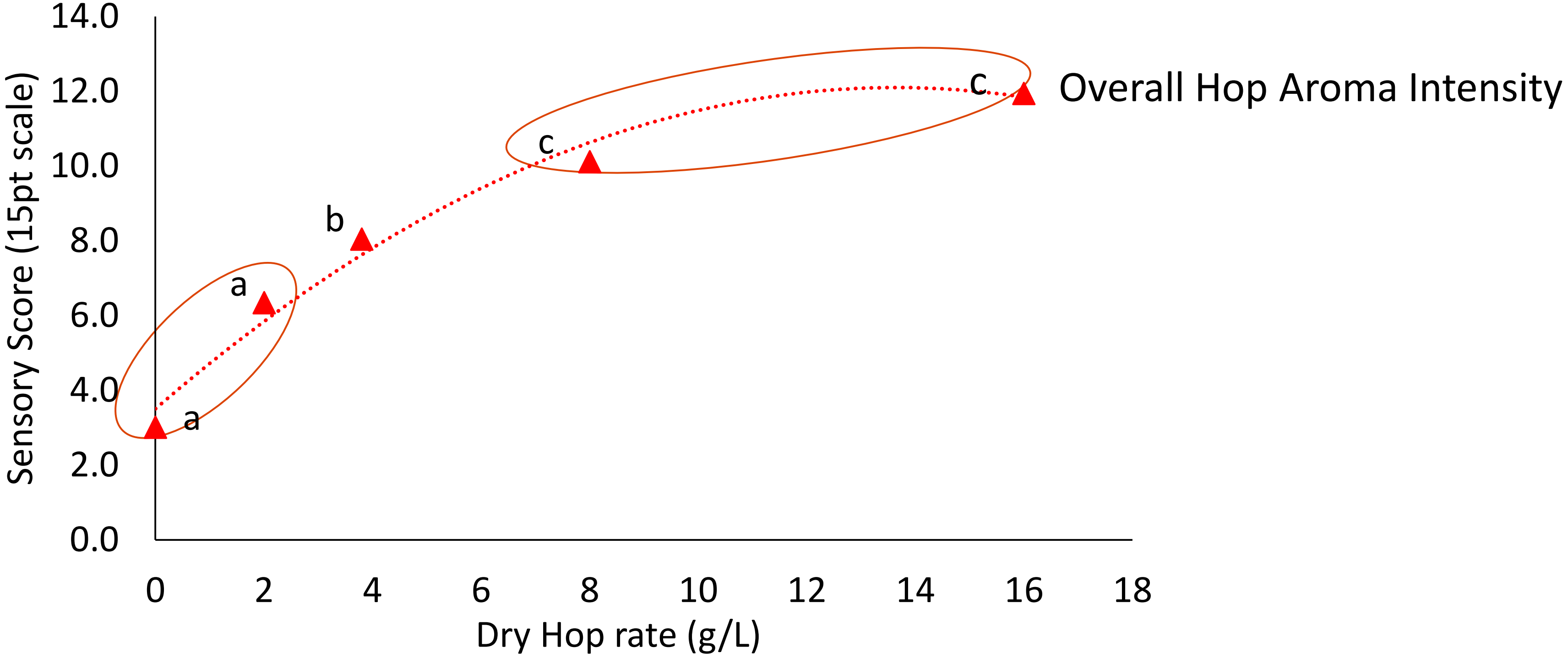
SENSORY RESULTS

Oregon State University

Hop dosage rate – Overall Hop Aroma Intensity



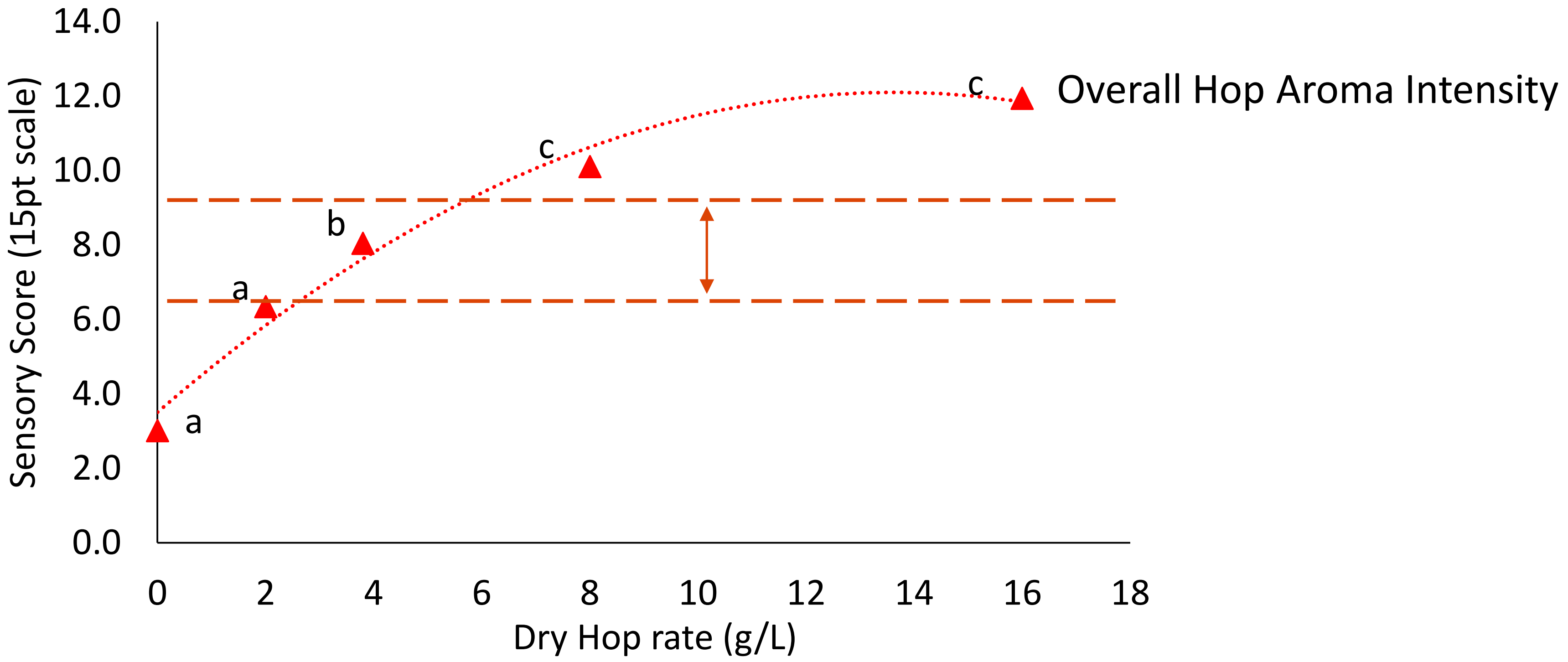
Hop dosage rate – Overall Hop Aroma Intensity



- **Panelists could discriminate the different dry hop rate samples (ie 0, 2, 3.8, 8, & 16)**

*Letters represent Tukey's HSD groupings

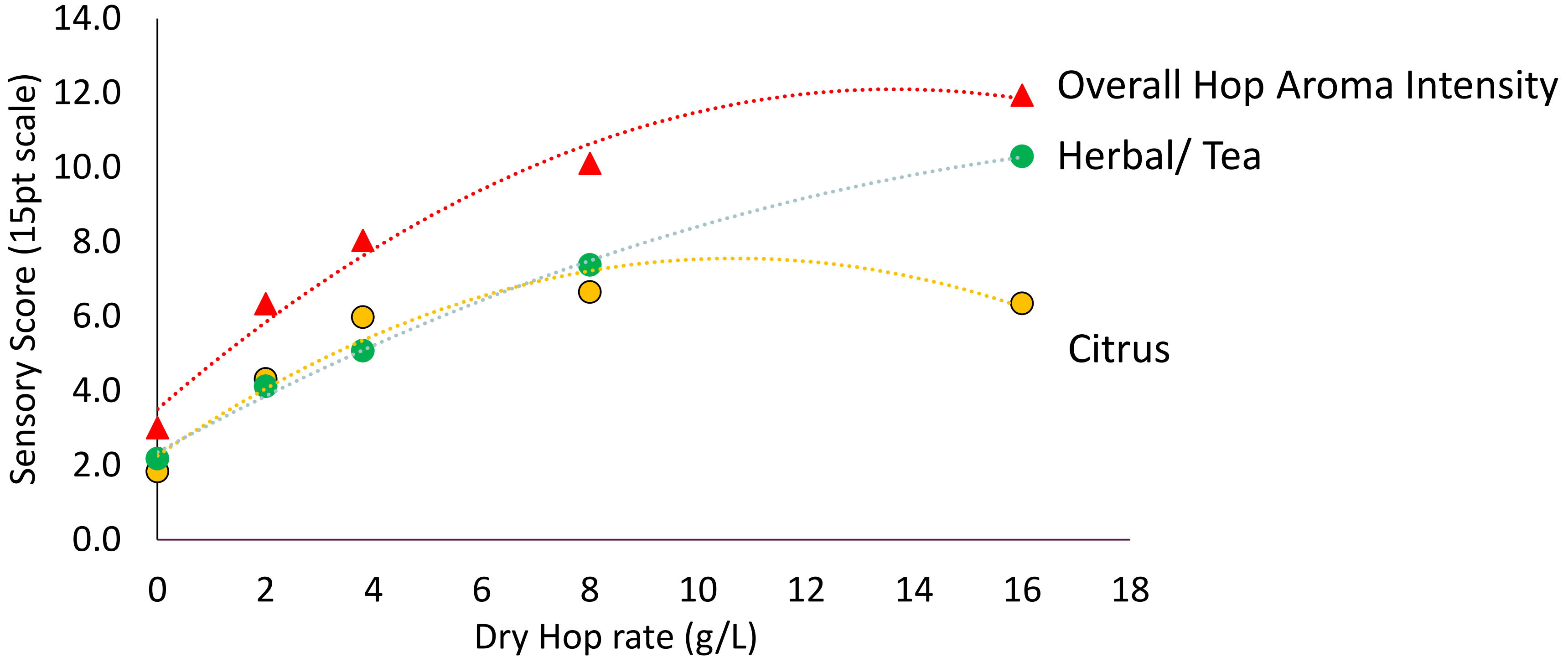
Hop dosage rate – Overall Hop Aroma Intensity



- **Panelists scaled the samples randomly against 28 other samples in the DA panel**
- **Range of DA OHAI ratings (i.e. ~6- 9.5) for 2015 DA Panels –Same DH rate 3.8/g/L**

*Letters represent Tukey's HSD groupings

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



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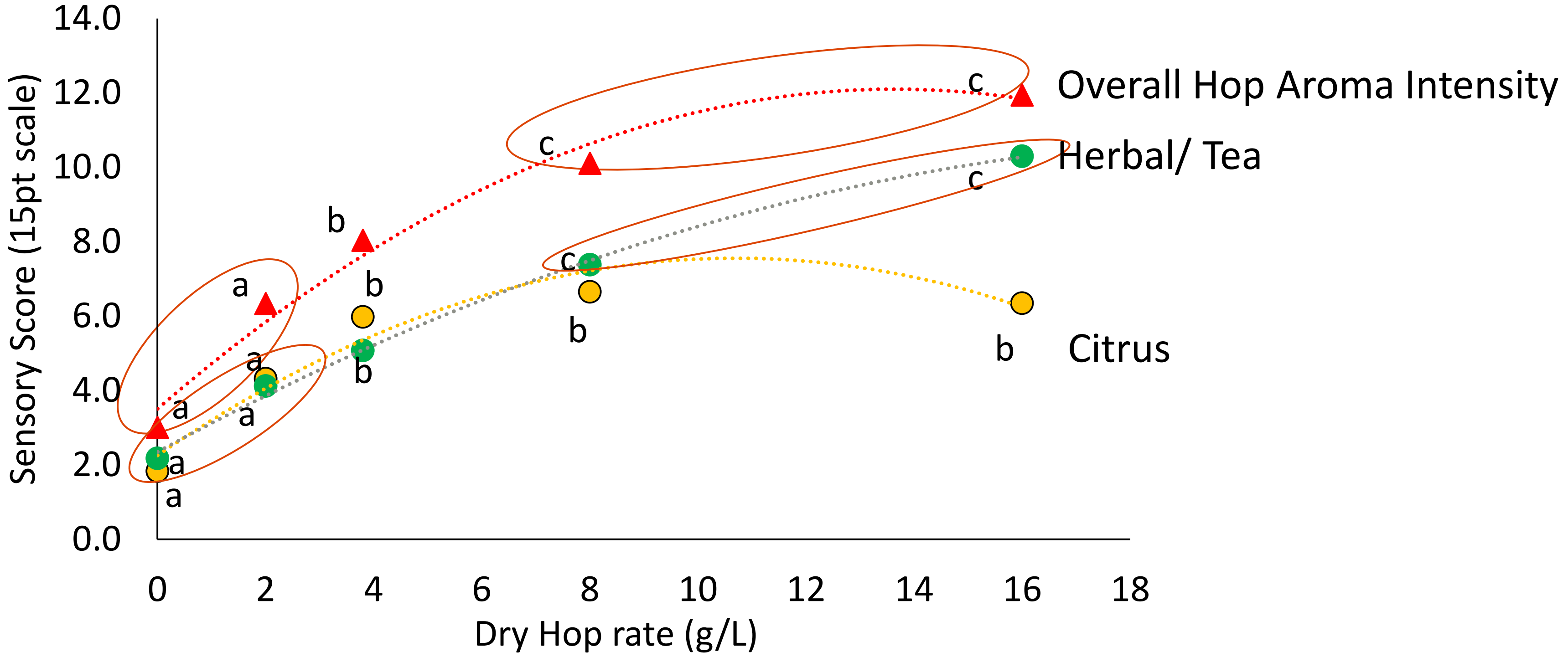

- **Herbal/ Tea response is similar to OHAI**

*Letters represent Tukey's HSD groupings

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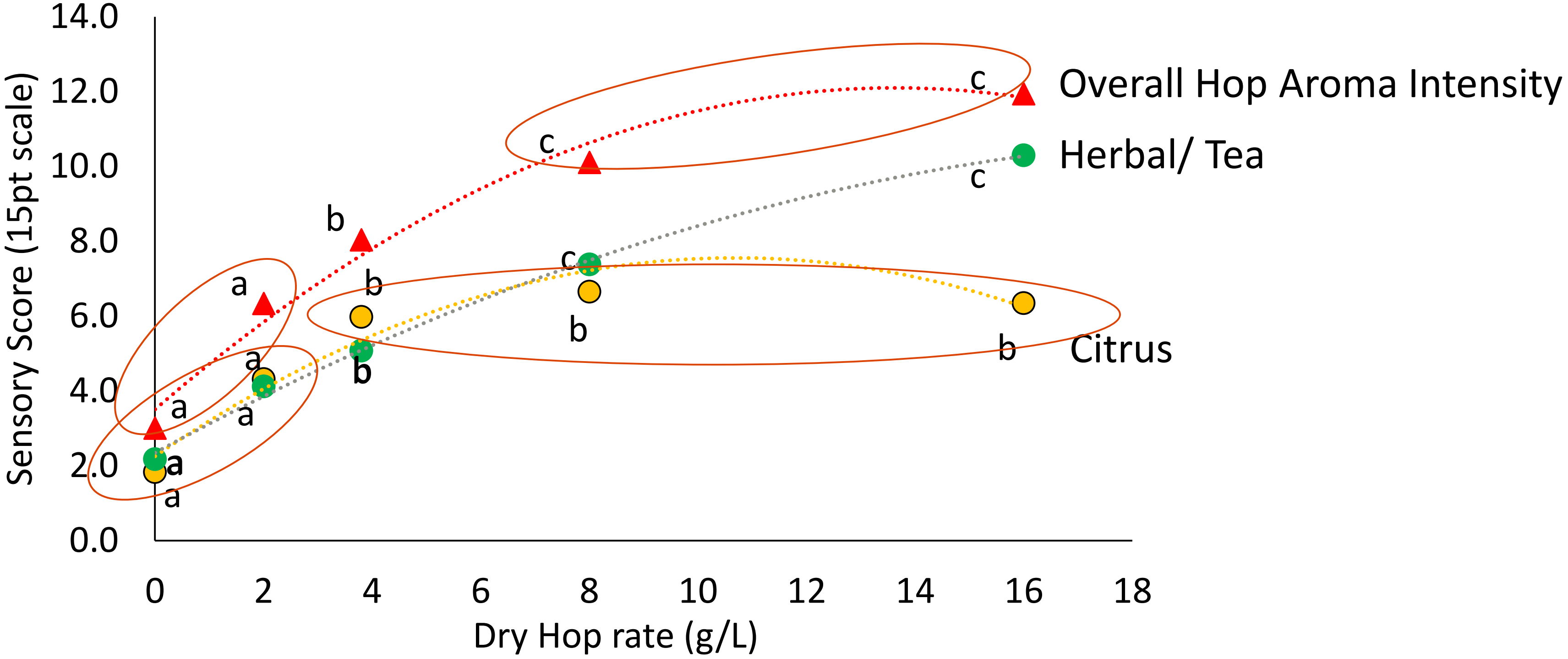
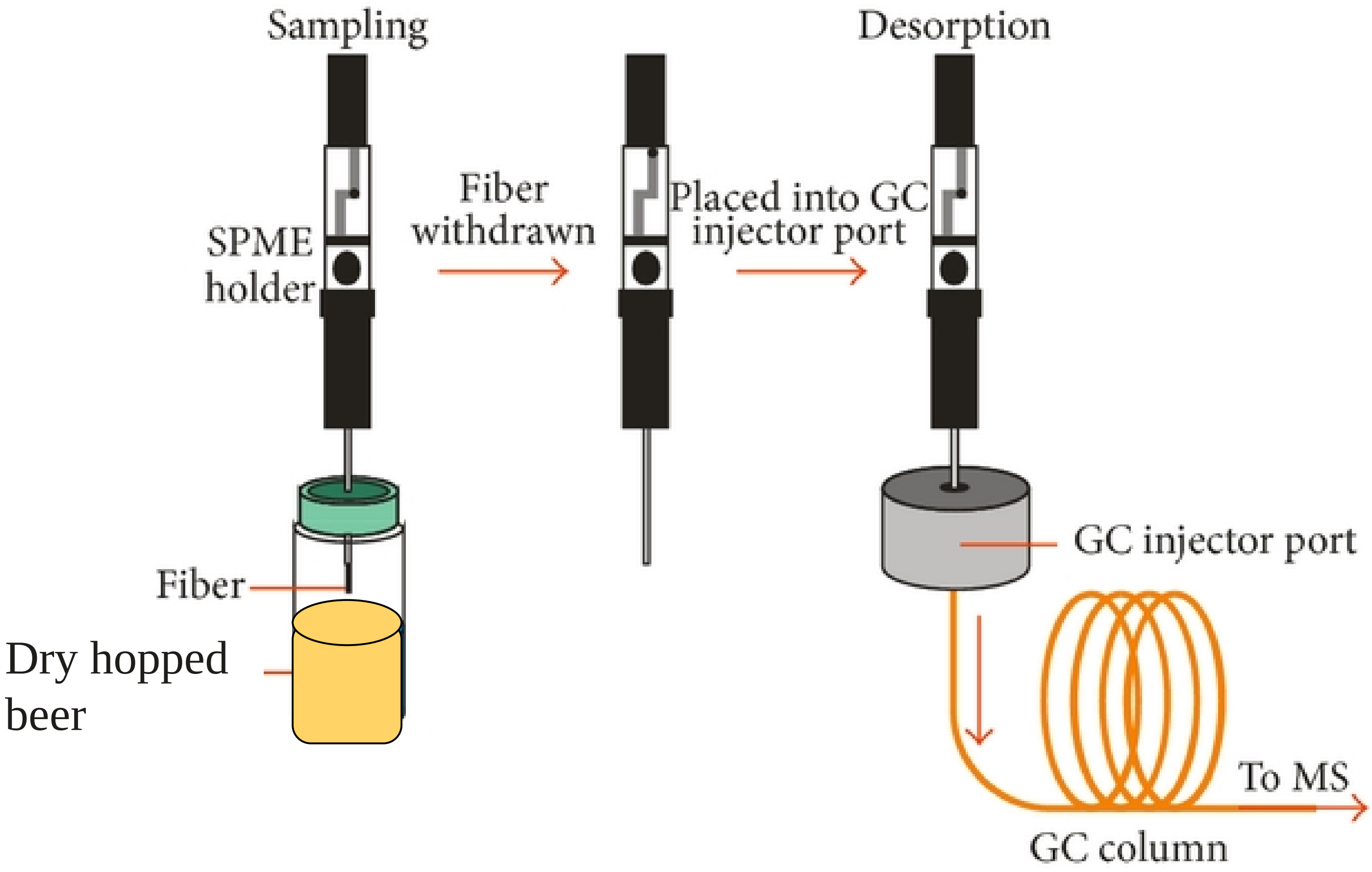


- **Citrus response seemed to be suppressed compared to OHAI and Herbal/Tea**

*Letters represent Tukey's HSD groupings

CHEMISTRY RESULTS

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

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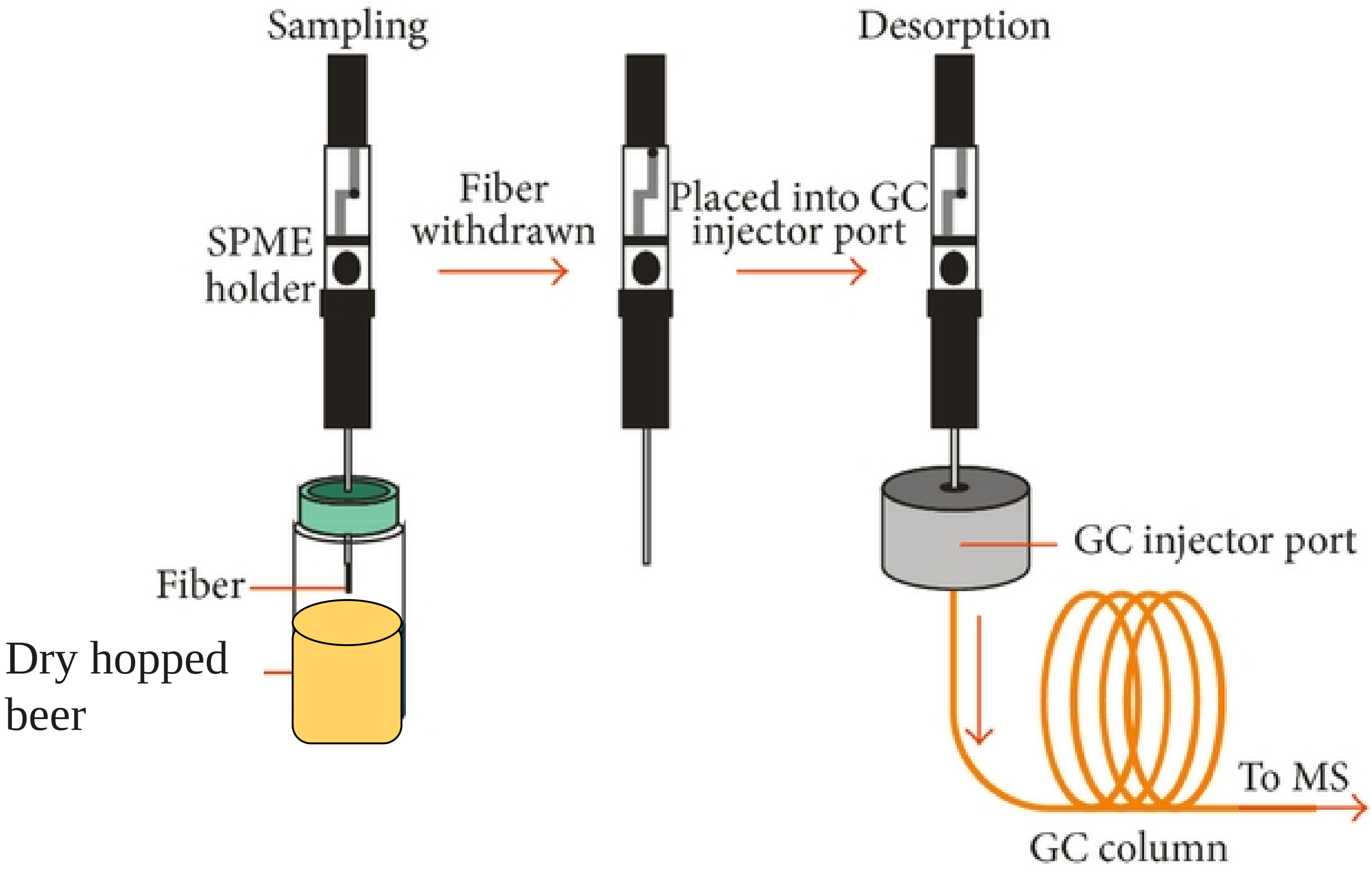

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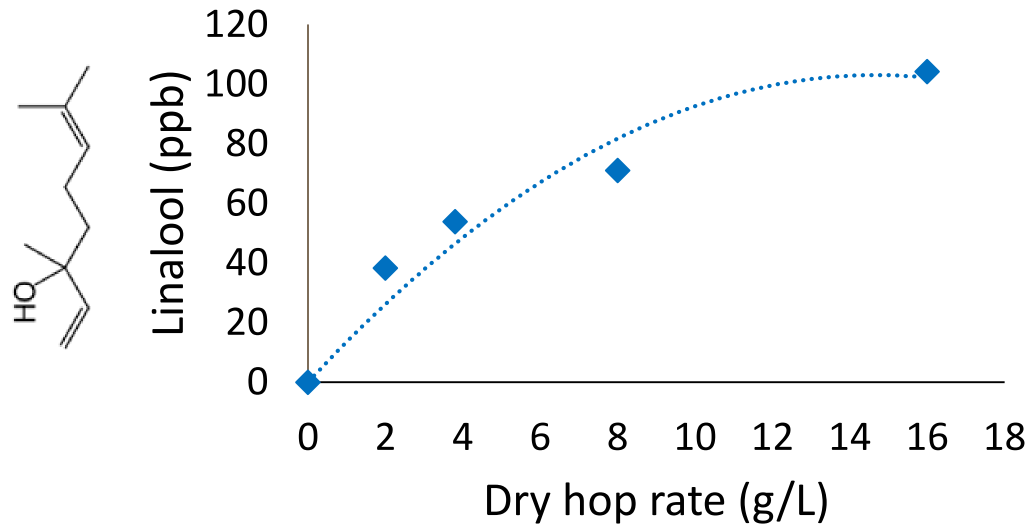
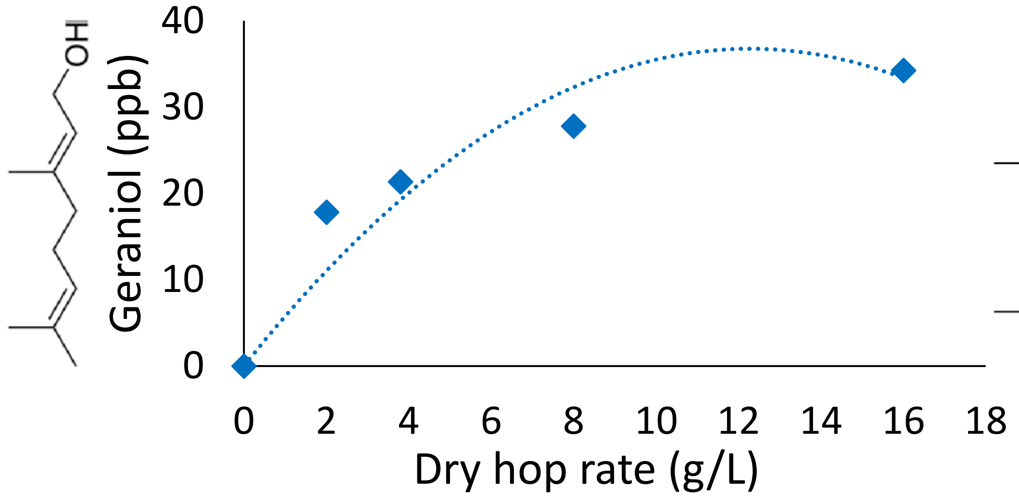
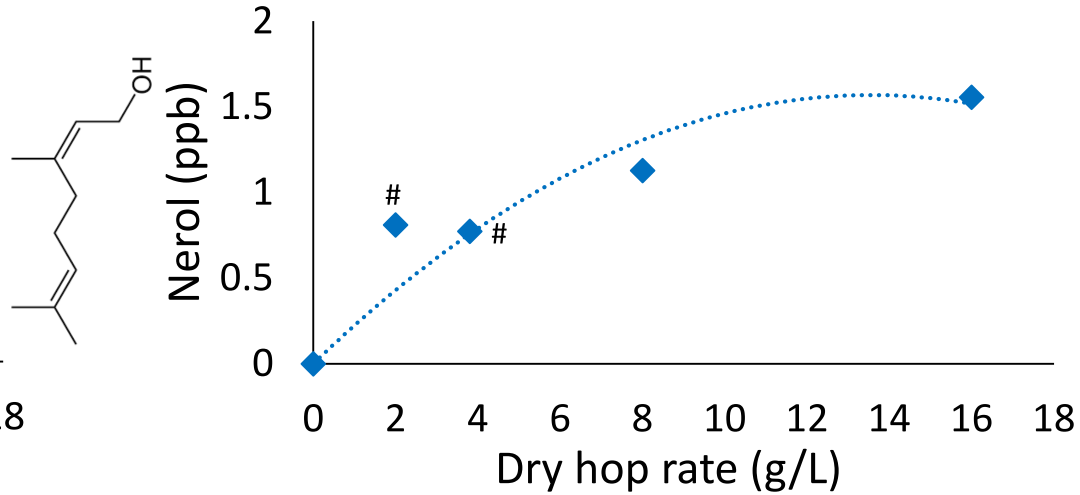
Hop Compound Concentrations – Terpene Alcohols



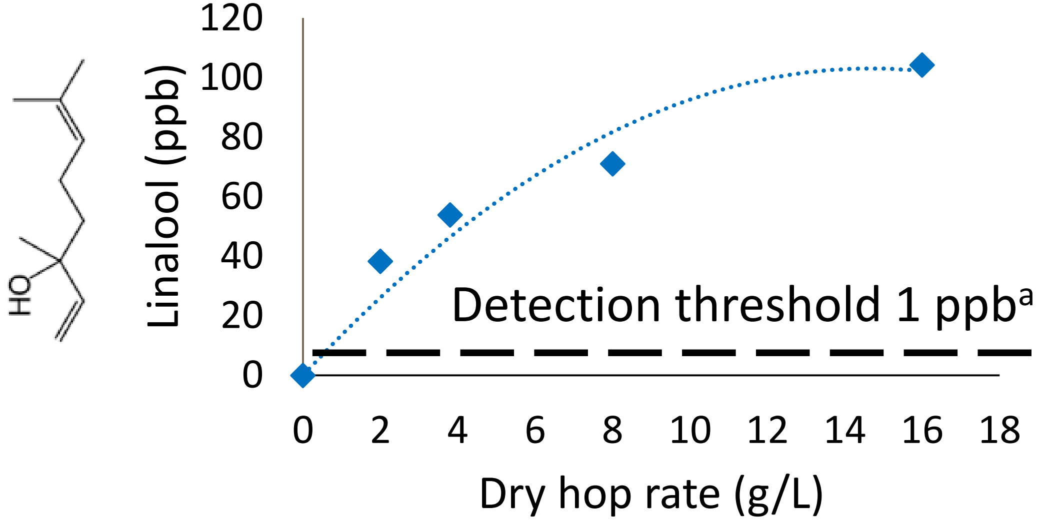




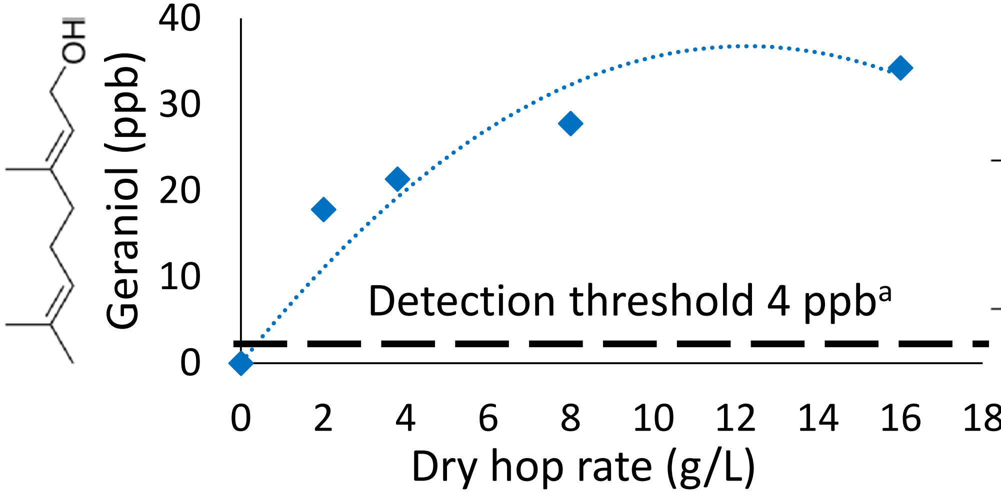
*Average of 4 SPME-GC/MS instrumental runs

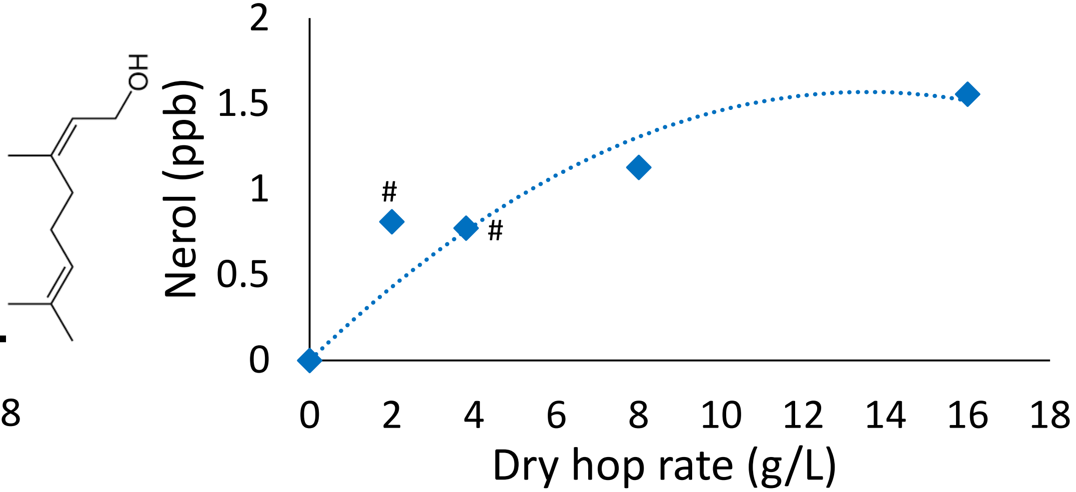
#Estimated values lower than LOQ

Hop Compound Concentrations – Terpene Alcohols



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

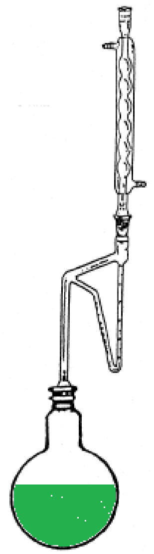


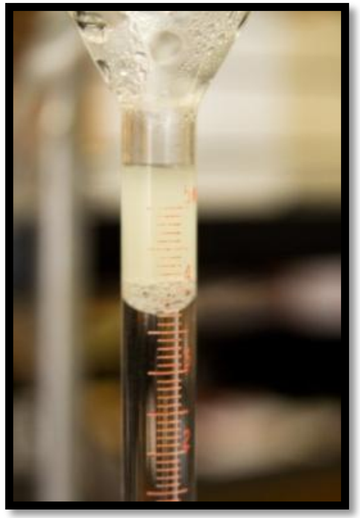


*Average of 4 SPME-GC/MS instrumental runs

#Estimated values lower than LOQ

Hop total oil + compositional analysis





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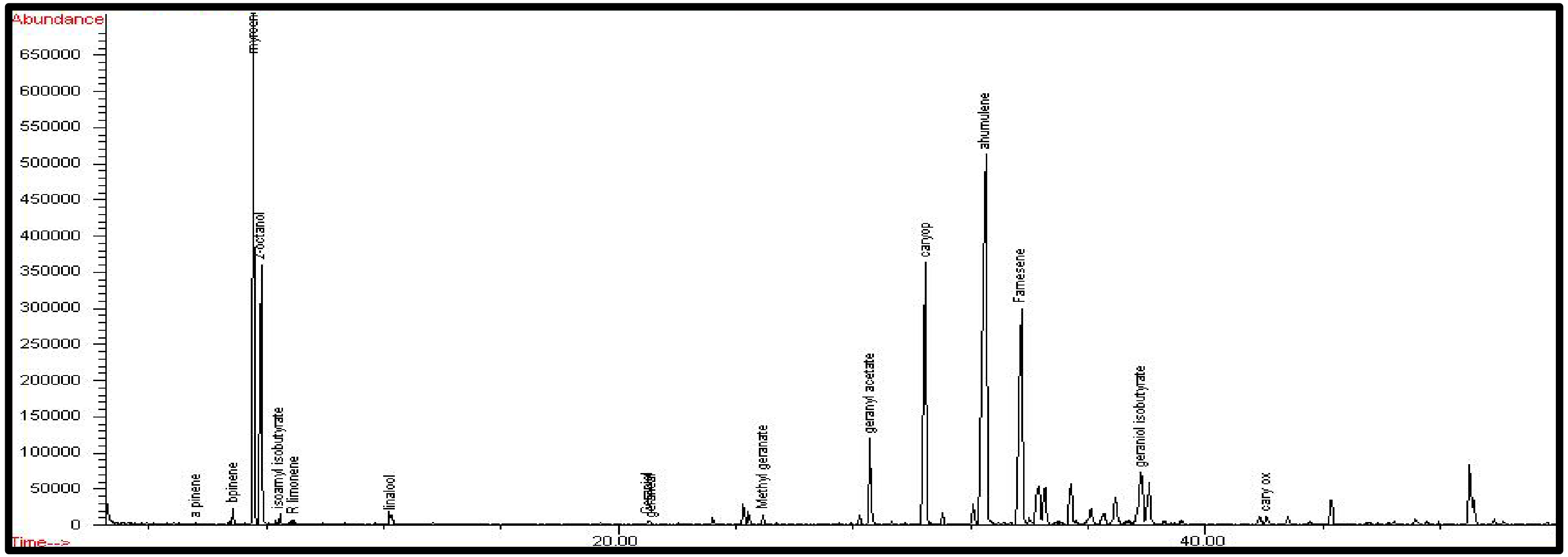
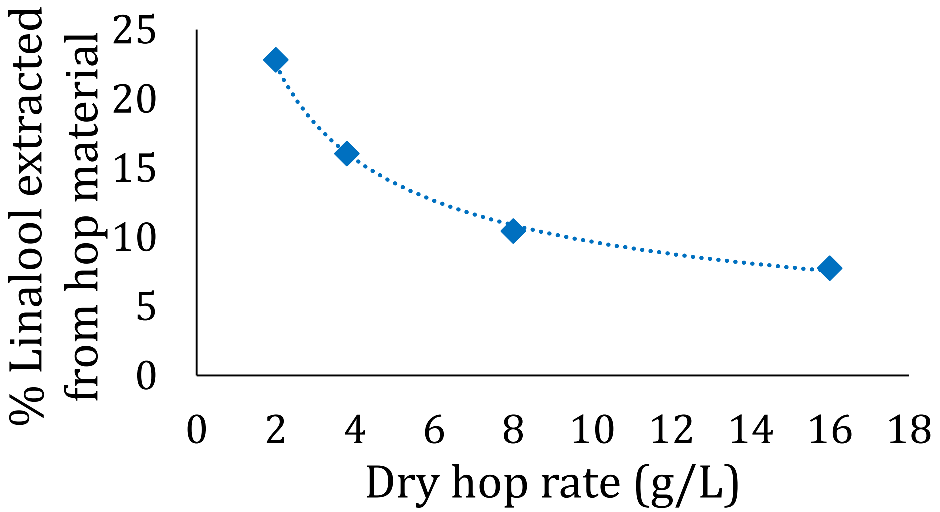
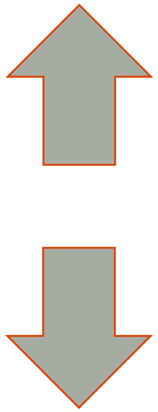


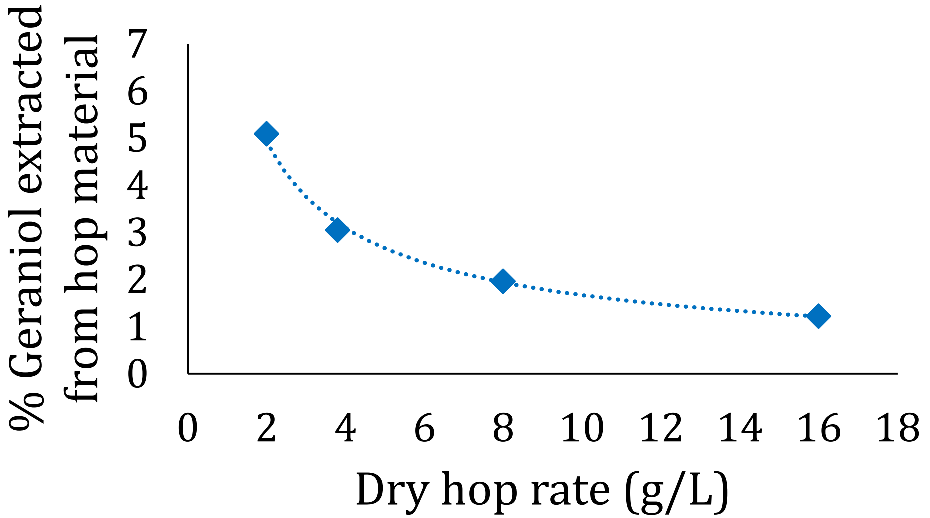
Hop Compound % Extraction – Terpene Alcohols

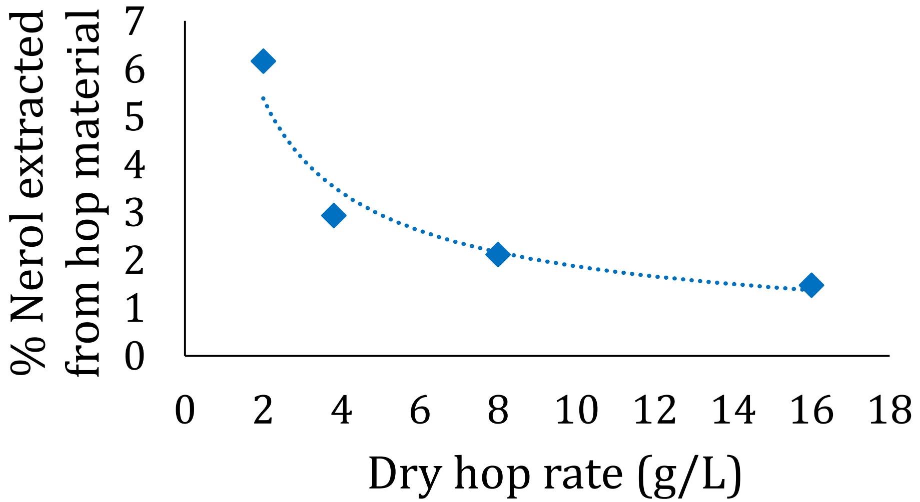




Dry hop rate

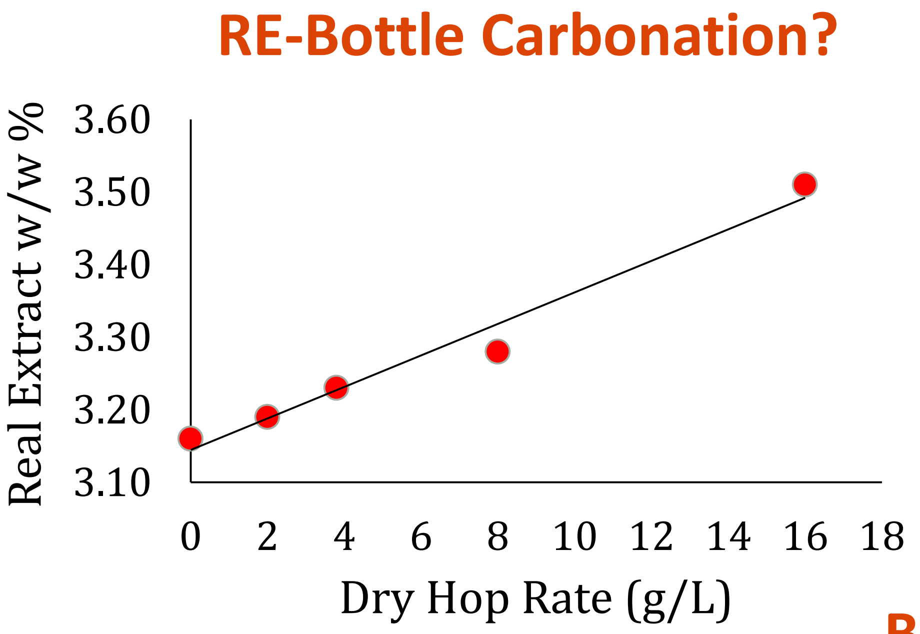
Terpene alcohol extraction (%)

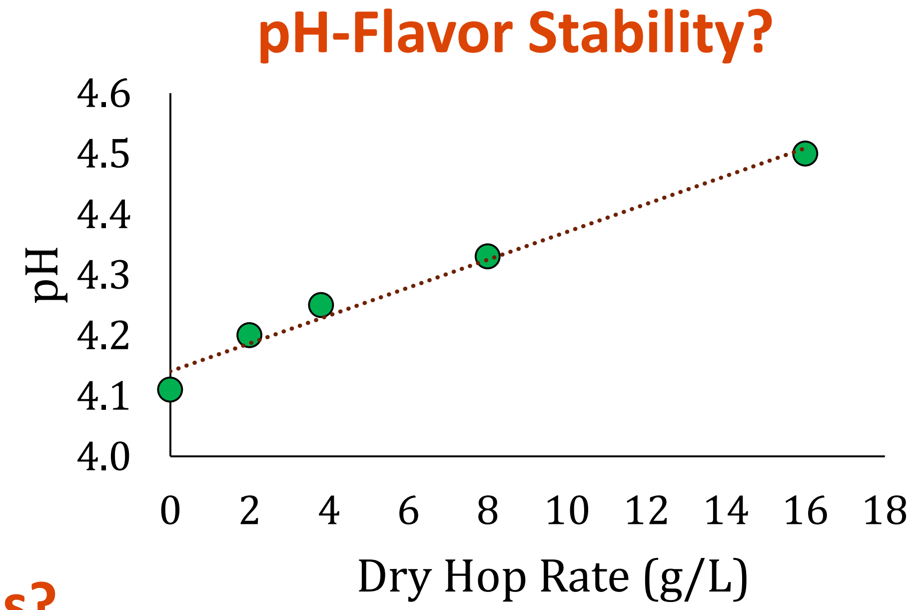


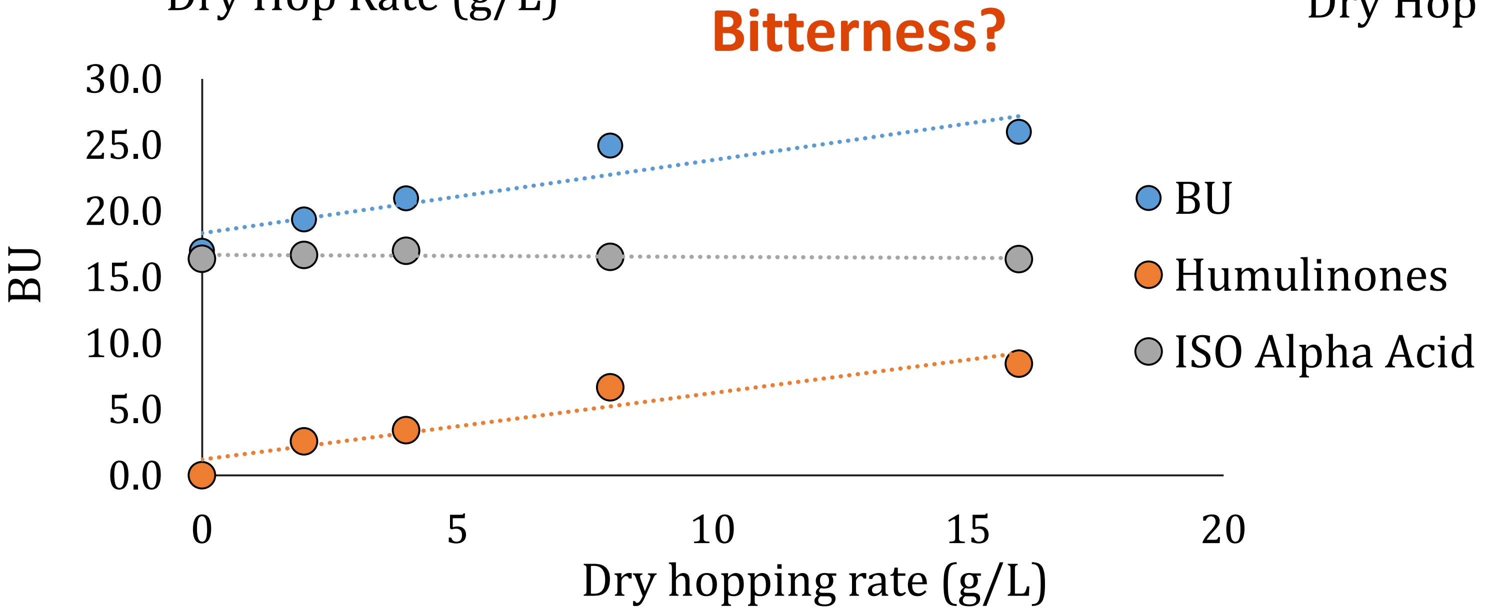


*Assuming 100% extraction based on hop oil hydrodistillation

Dry hopping having other impacts on beer……







*pH and RE measured using Anton Paar Alcolyzer

*BU measured using ASBC MOA Beer-23

Conclusions

- More hops is not an efficient way of adding aroma to beer
  - Soon to be published.. Impact of static dry-hopping rate on the sensory and analytical profiles of beer

- *Can spent dry-hops be reused?*
  - ***Look for Dean Hauser's work at Brewing Summit-*** *"The Extraction Efficiency of Hop Bitter Acids and Volatiles During Dry-Hopping"*

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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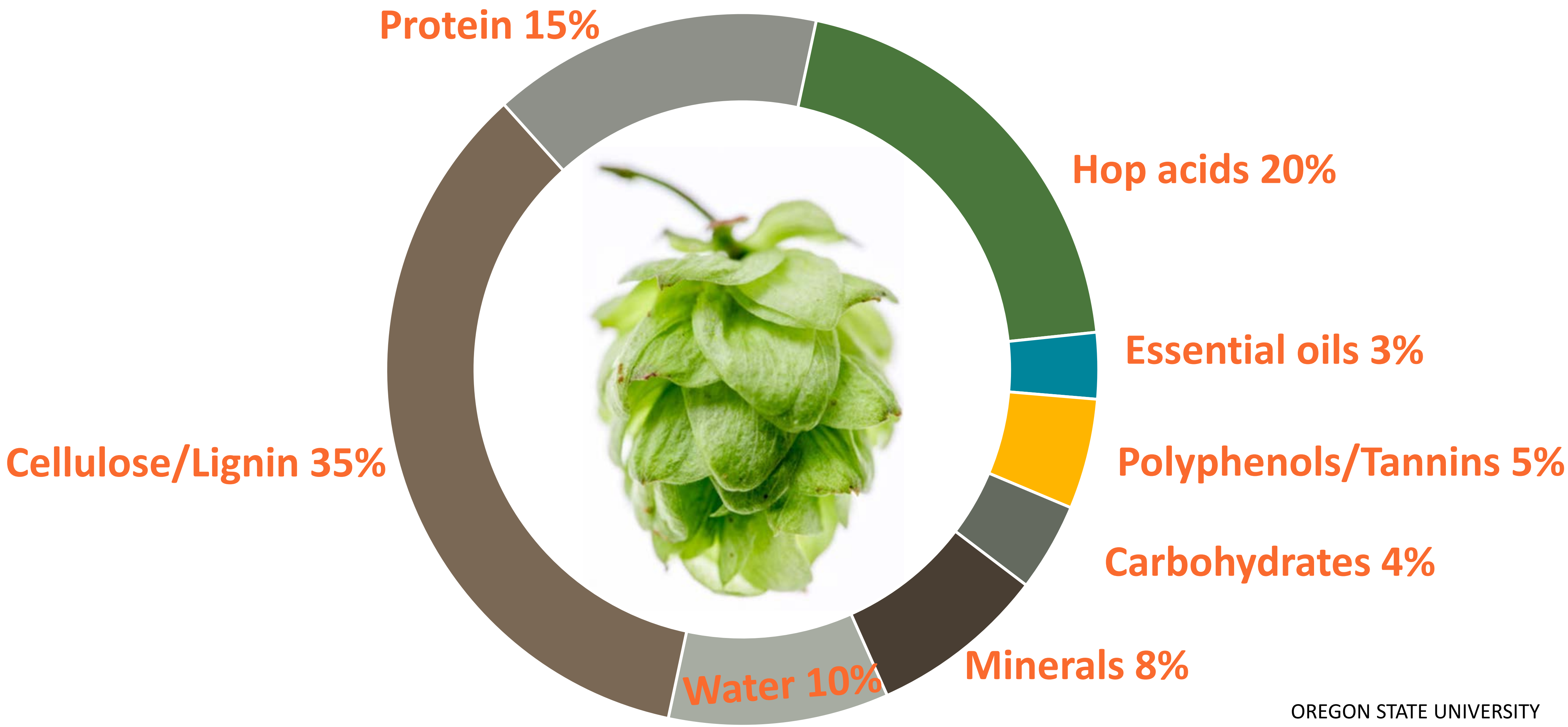
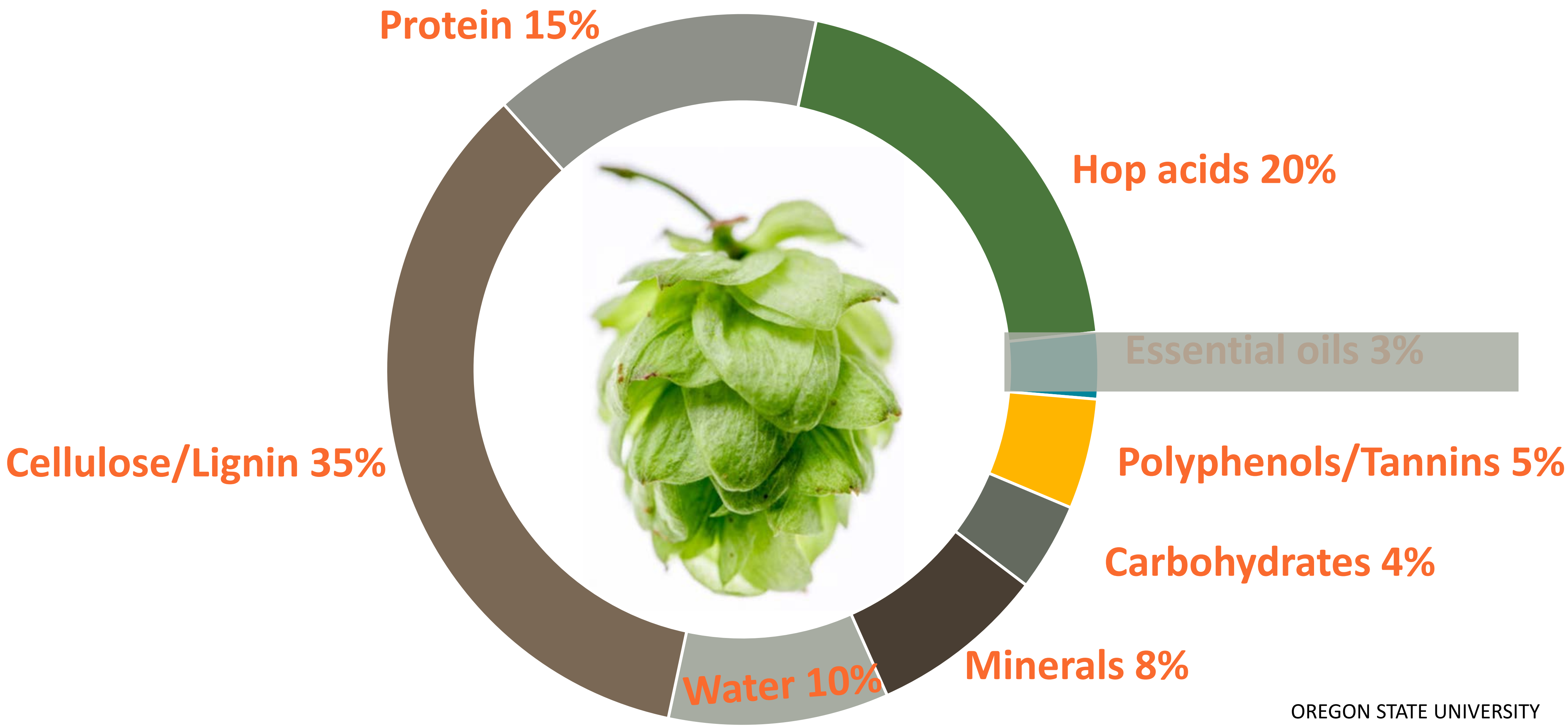


How does the nonvolatile fraction influence dry-hopped beer quality?



Hop acids (up to ~20% of hops)

Alpha Acids

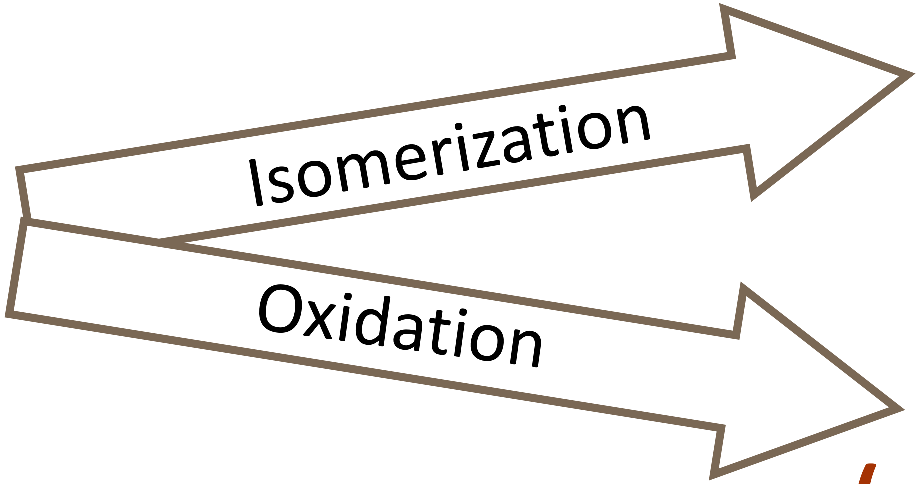
Beta Acids

Hop acids (up to ~20% of hops)

Alpha Acids $\xrightarrow{\text{Isomerization}}$ **Iso-Alpha Acids**

Hop acids (up to ~20% of hops)

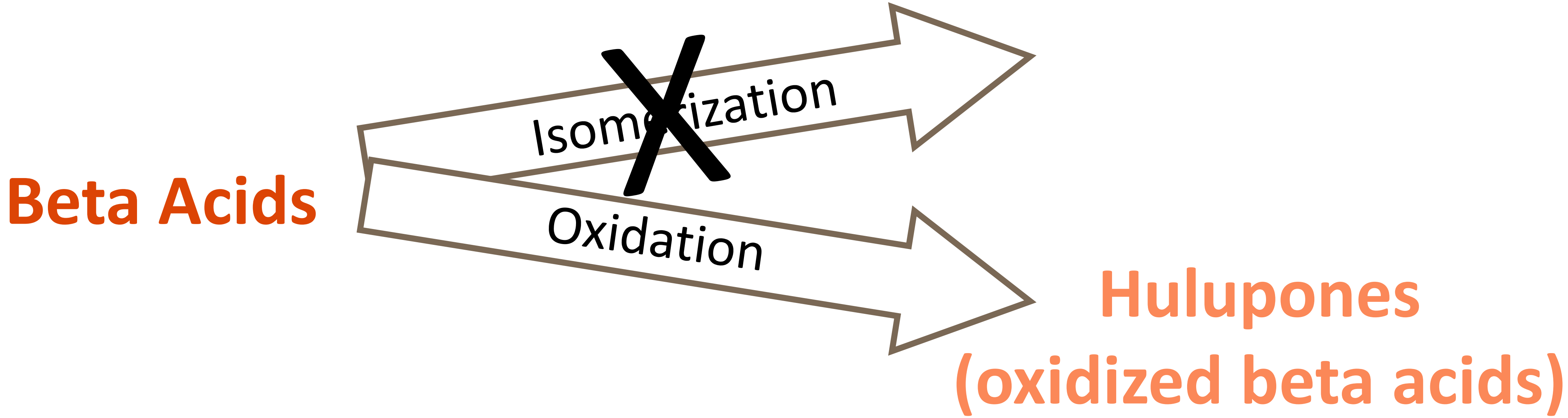
Alpha Acids



Iso-Alpha Acids

Humulinones
(oxidized alpha acids)

Hop acids (up to ~20% of hops)



Hop acids – what you may find in beer

Iso-Alpha Acids

Alpha Acids

Humulinones (oxidized alpha acids)

Hulupones (oxidized beta acids)

Hop acids – what you may find in beer

Iso-Alpha Acids

Alpha Acids

Humulinones (oxidized alpha acids)

~~Hulupones (oxidized beta acids)~~

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

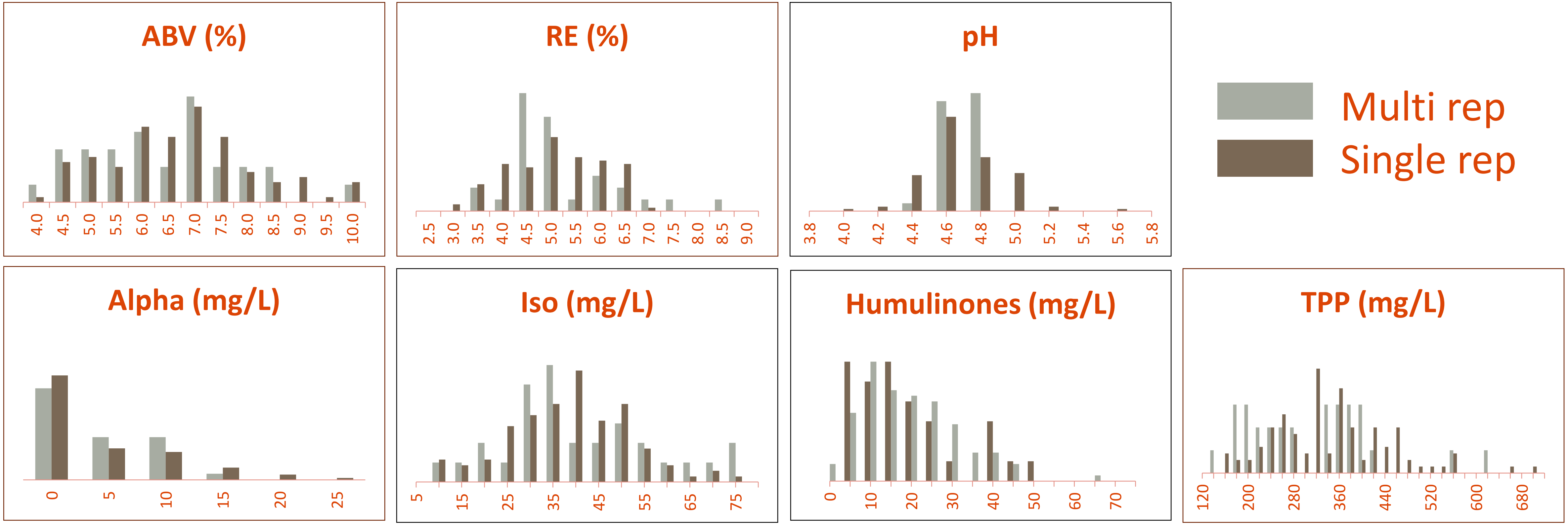
Chemical analysis: 7 factors

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

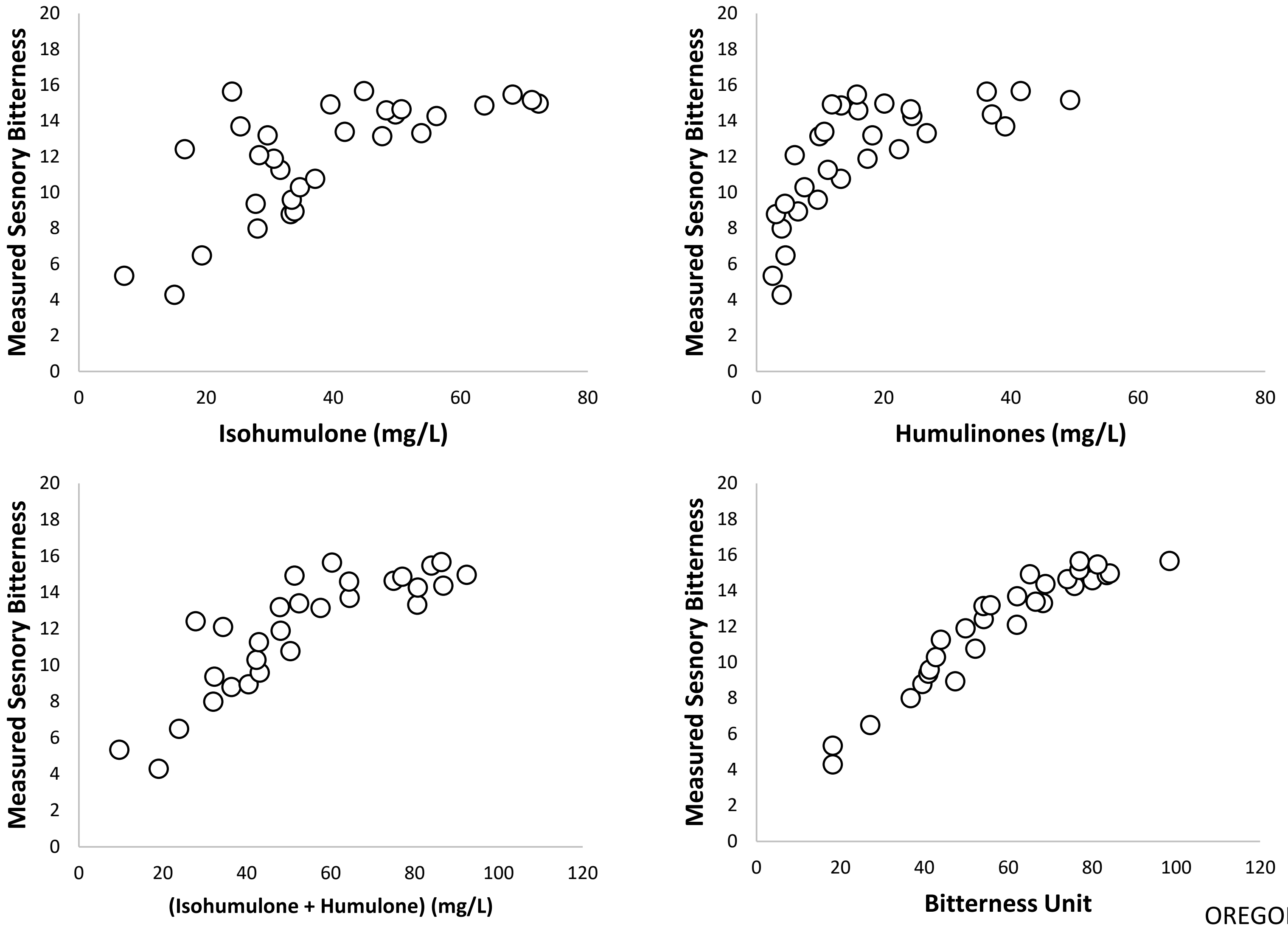
Sensory analysis:

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

Chemistry of beers in commercial survey



Bitterness comes from Isos & Humulinones
BU predicts bitterness



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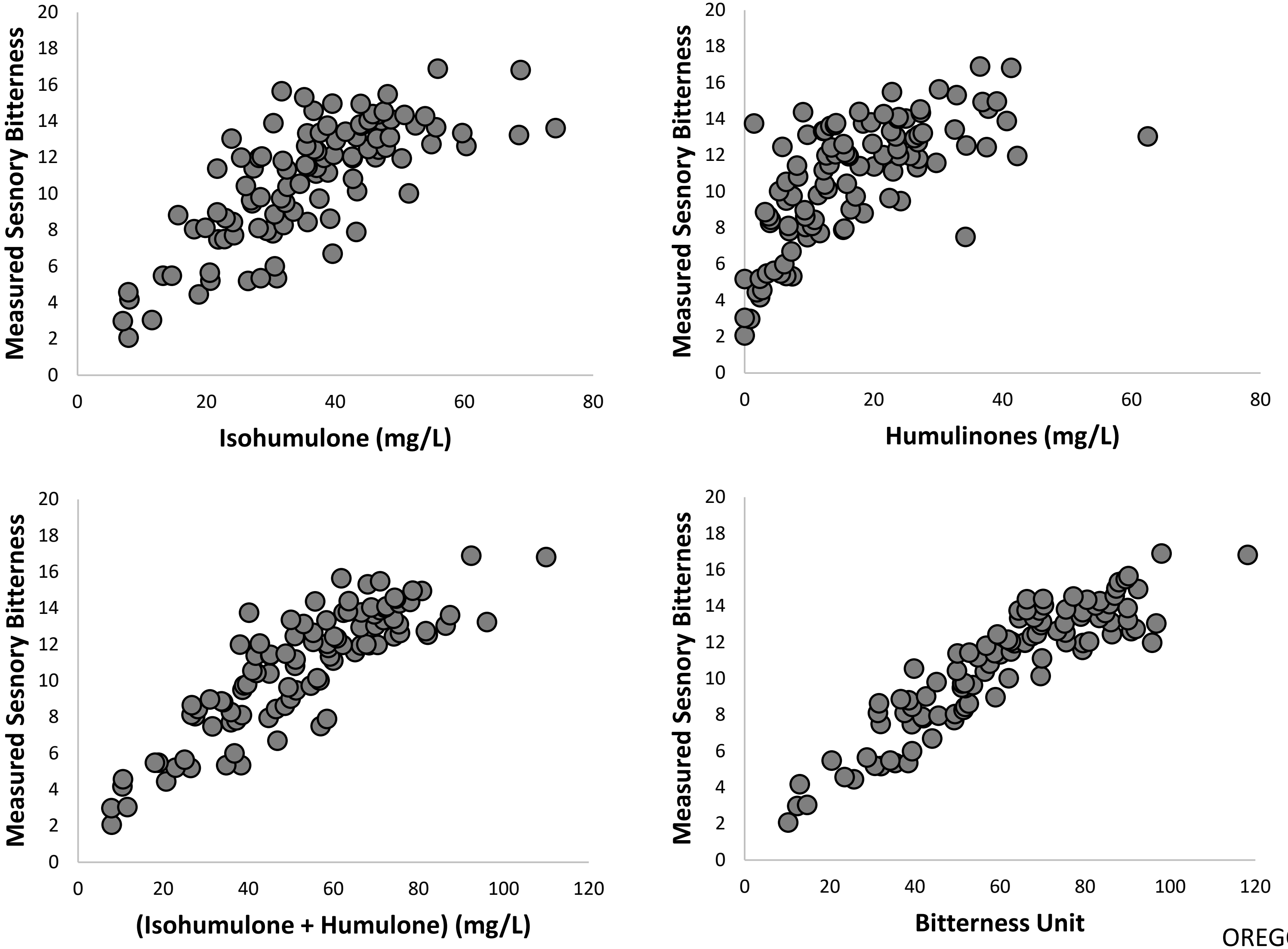

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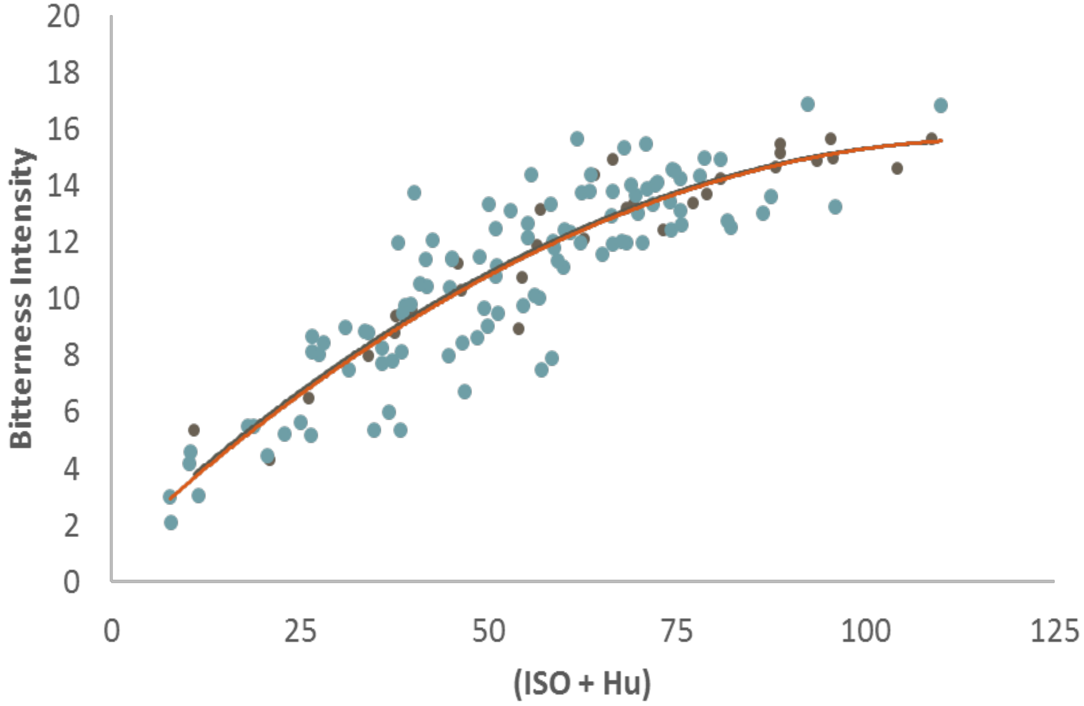
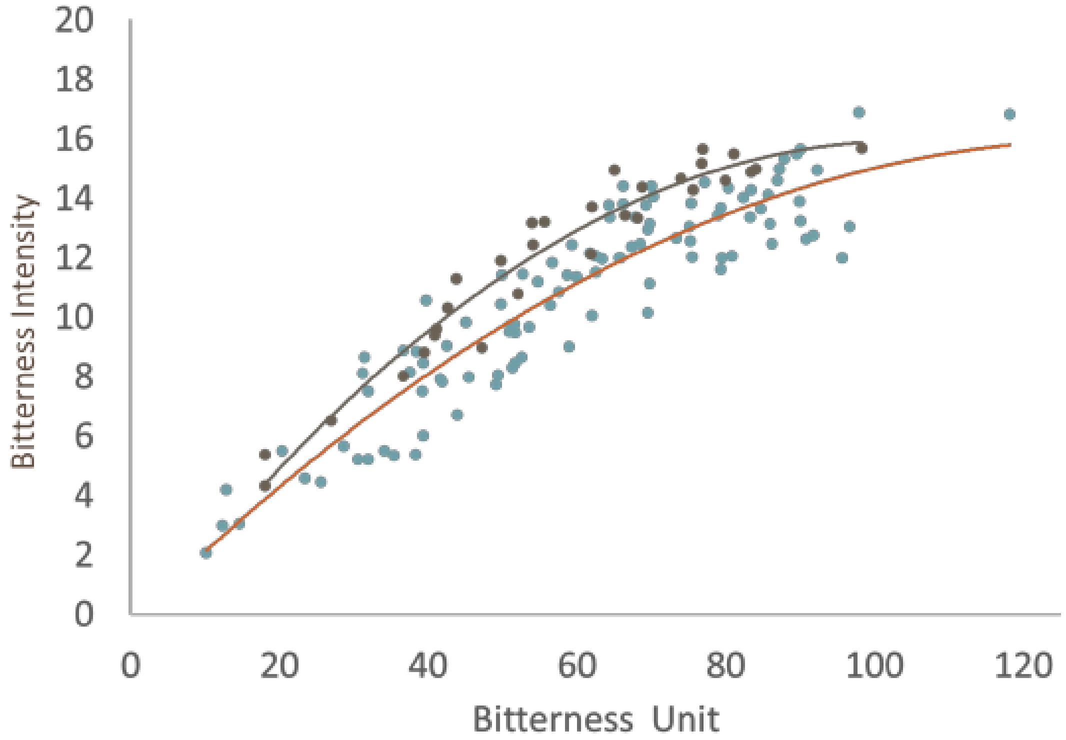


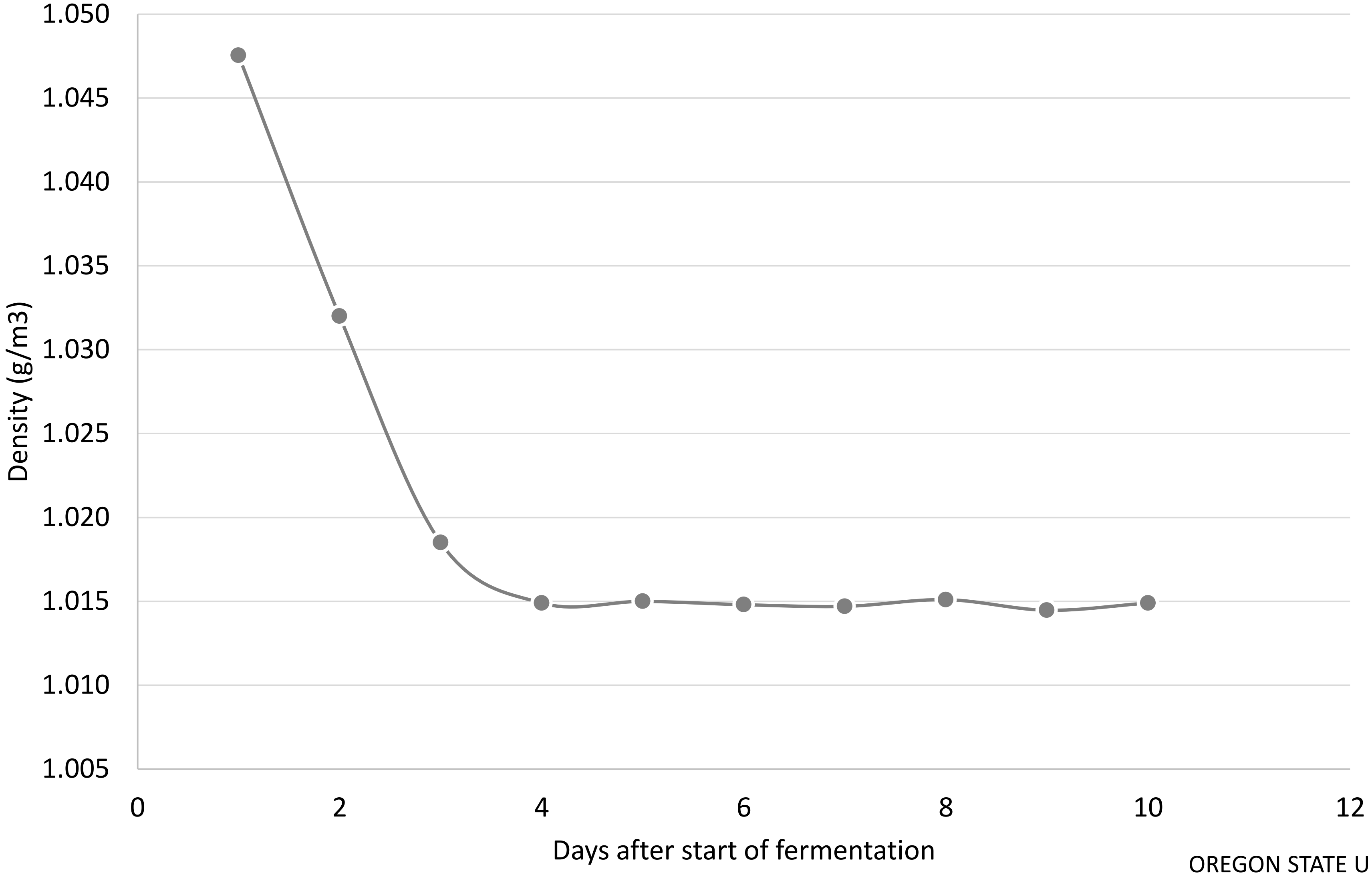


Hahn, C, Lafontaine, S.R., Pereira, C.B. and Shellhammer, T.H. 2018. Evaluation of the Nonvolatile Chemistry Affecting the Sensory Bitterness Intensity of Highly Hopped Beer, Journal of Agricultural and Food Chemistry

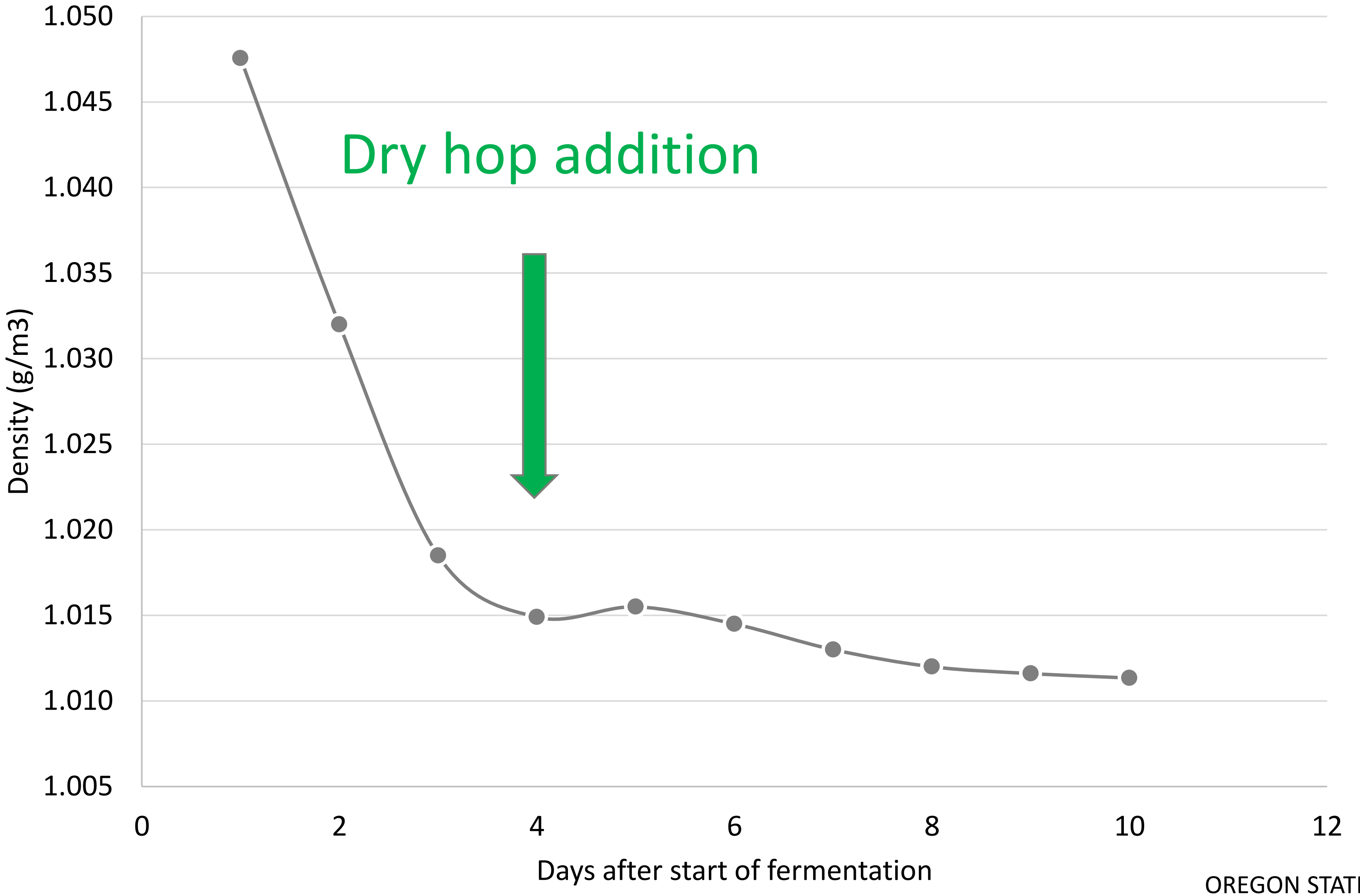
HOP CREEP

Oregon State University

Typical fermentation, no dry-hopping



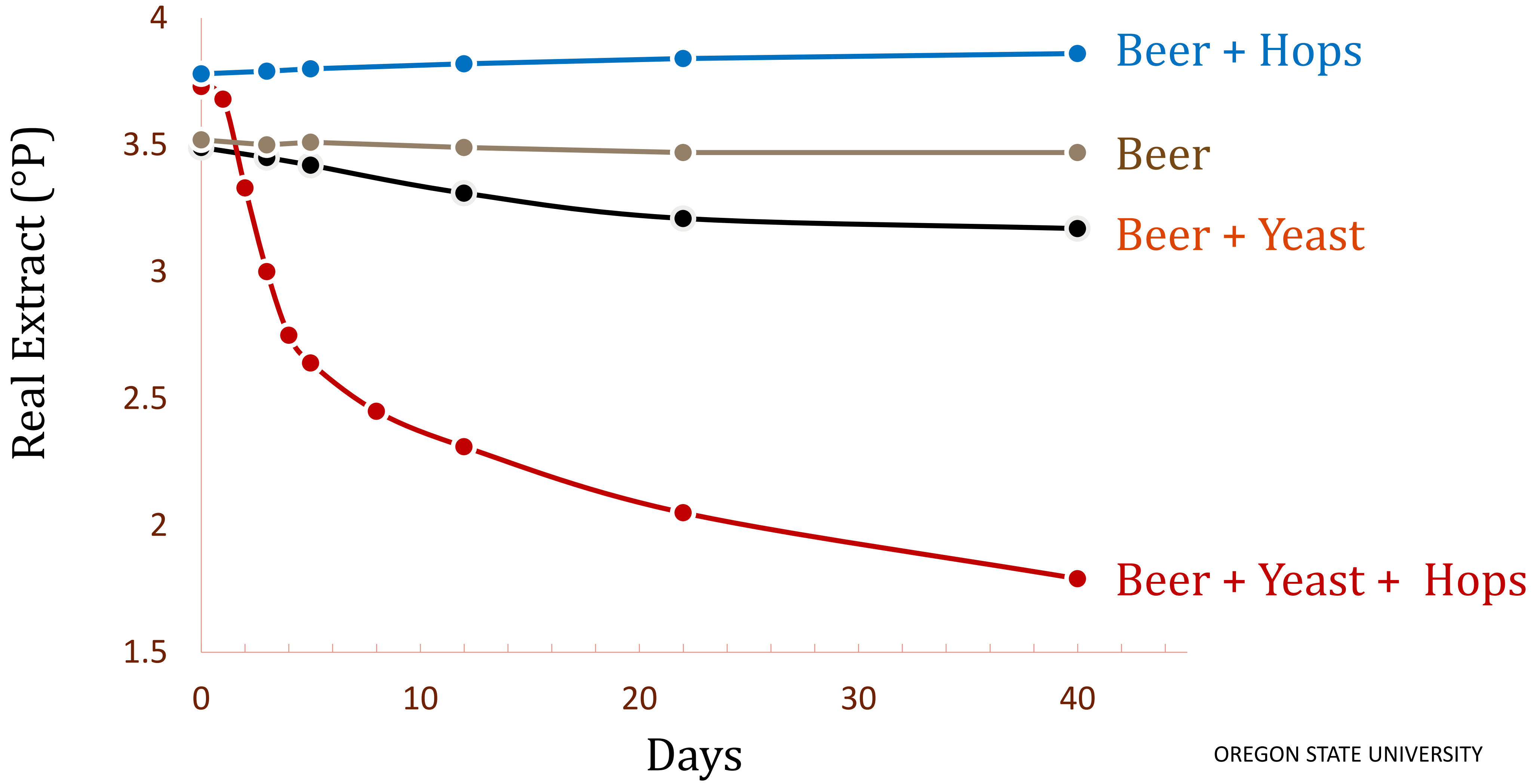
Dry-hopping can create "Hop Creep"



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

Hop enzymes stimulate “after-fermentation” AKA – Hop Creep



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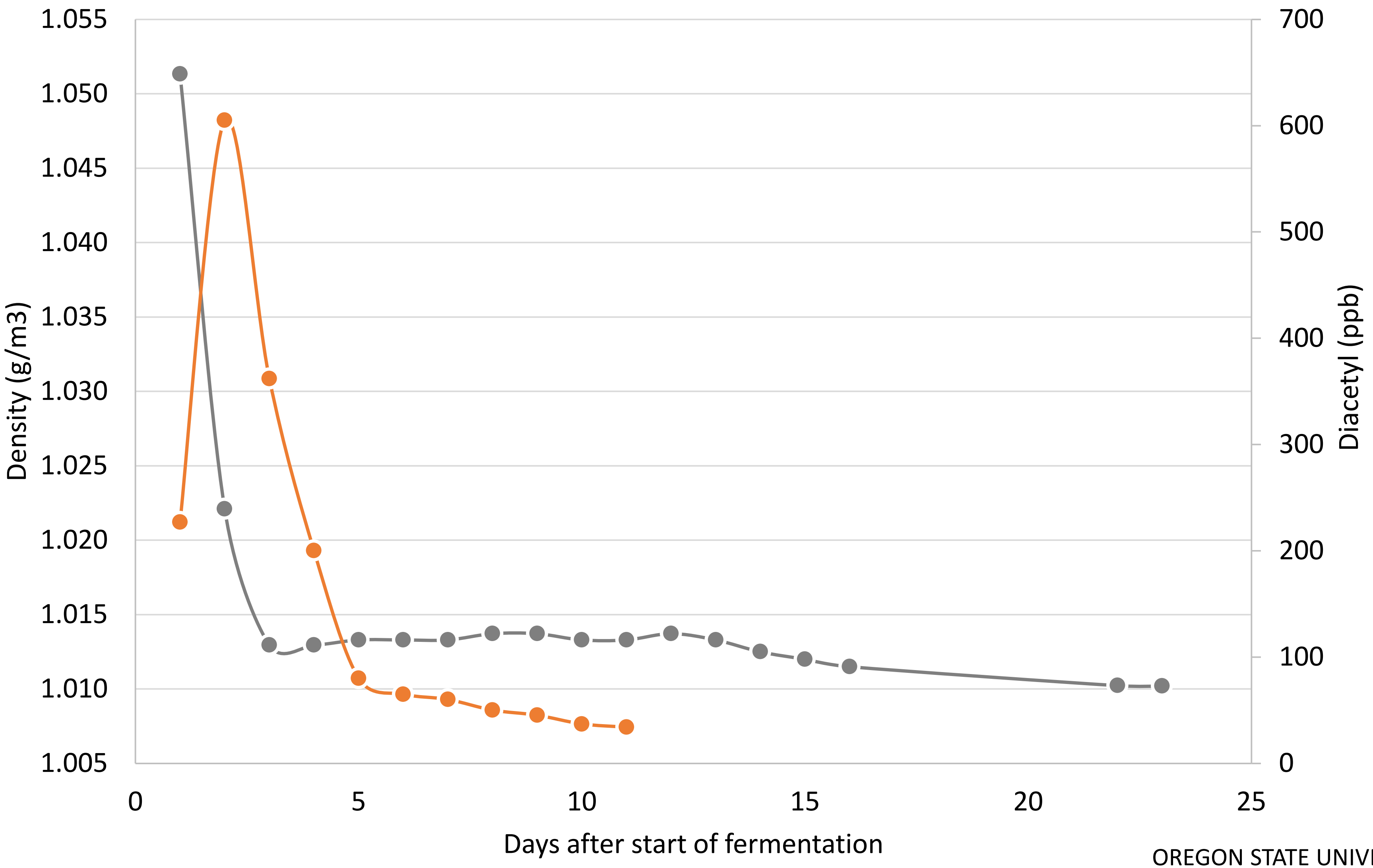
Hop Creep & Diacetyl issues



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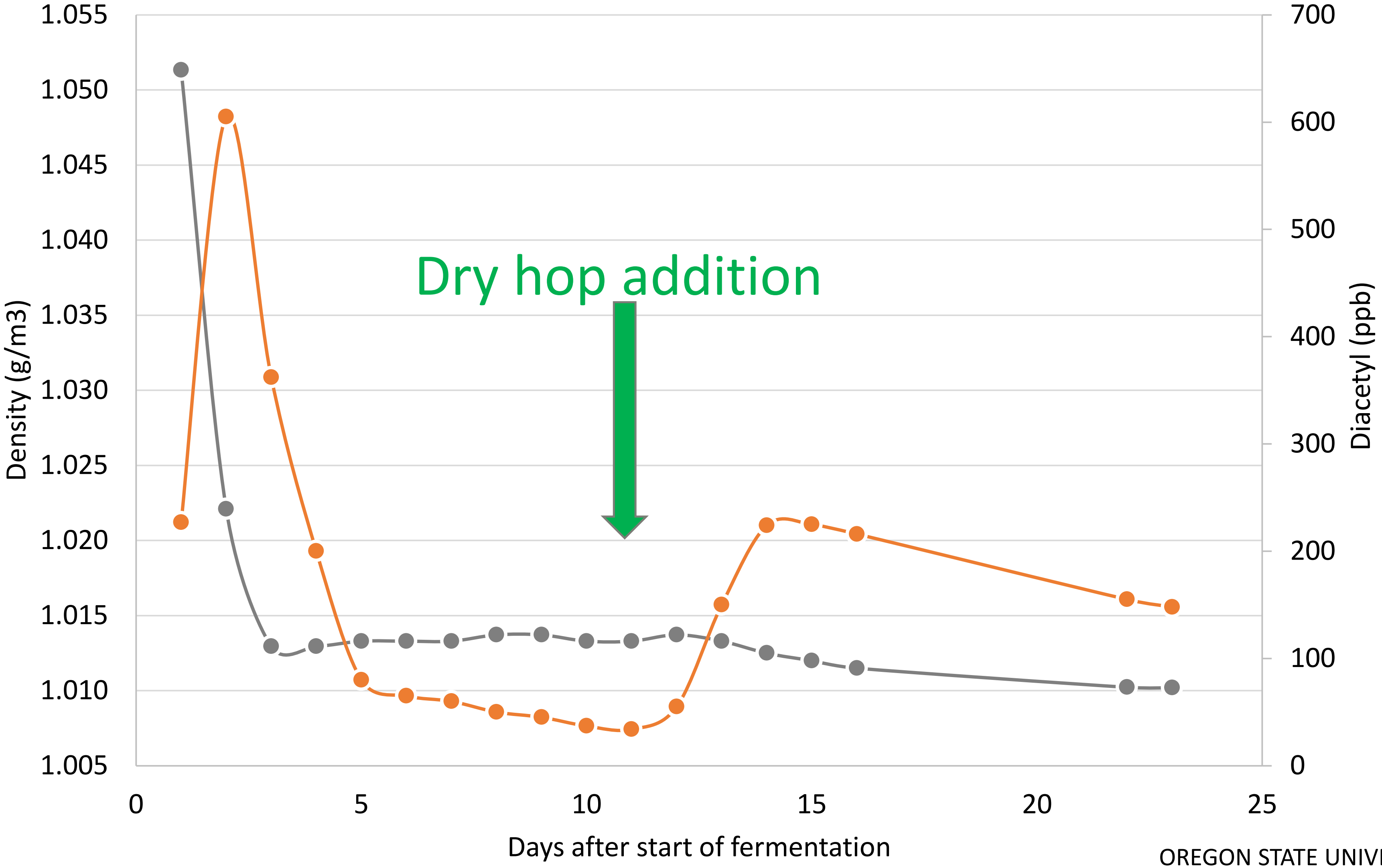
Hop Creep & Diacetyl issues



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

Sampling plan

Samples measured on Anton Paar Alcolyzer/Densitometer & HPLC

- Pre-dry hop addition
- Pre-second dry hop addition
- 24 hours
- 48 hours
- 72 hours

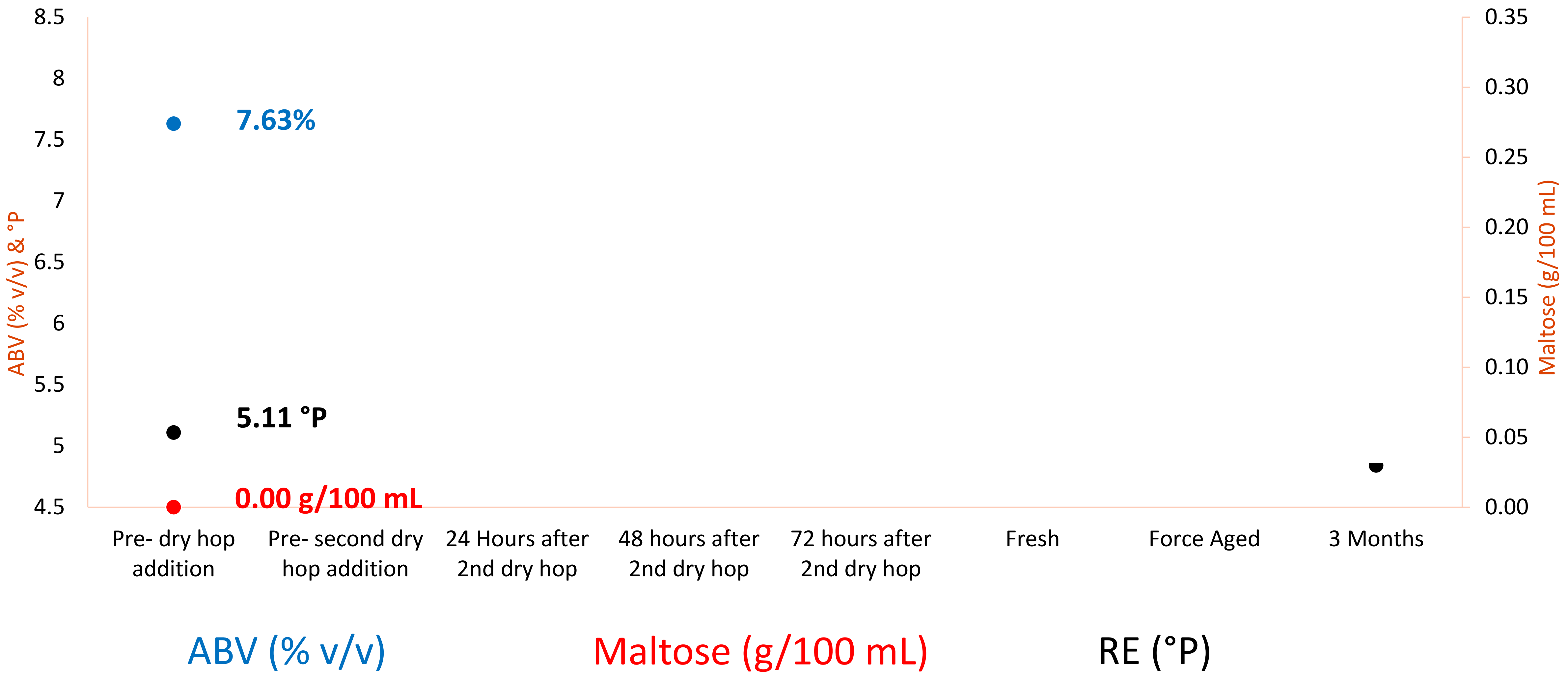
In-process samples

Finished beer samples

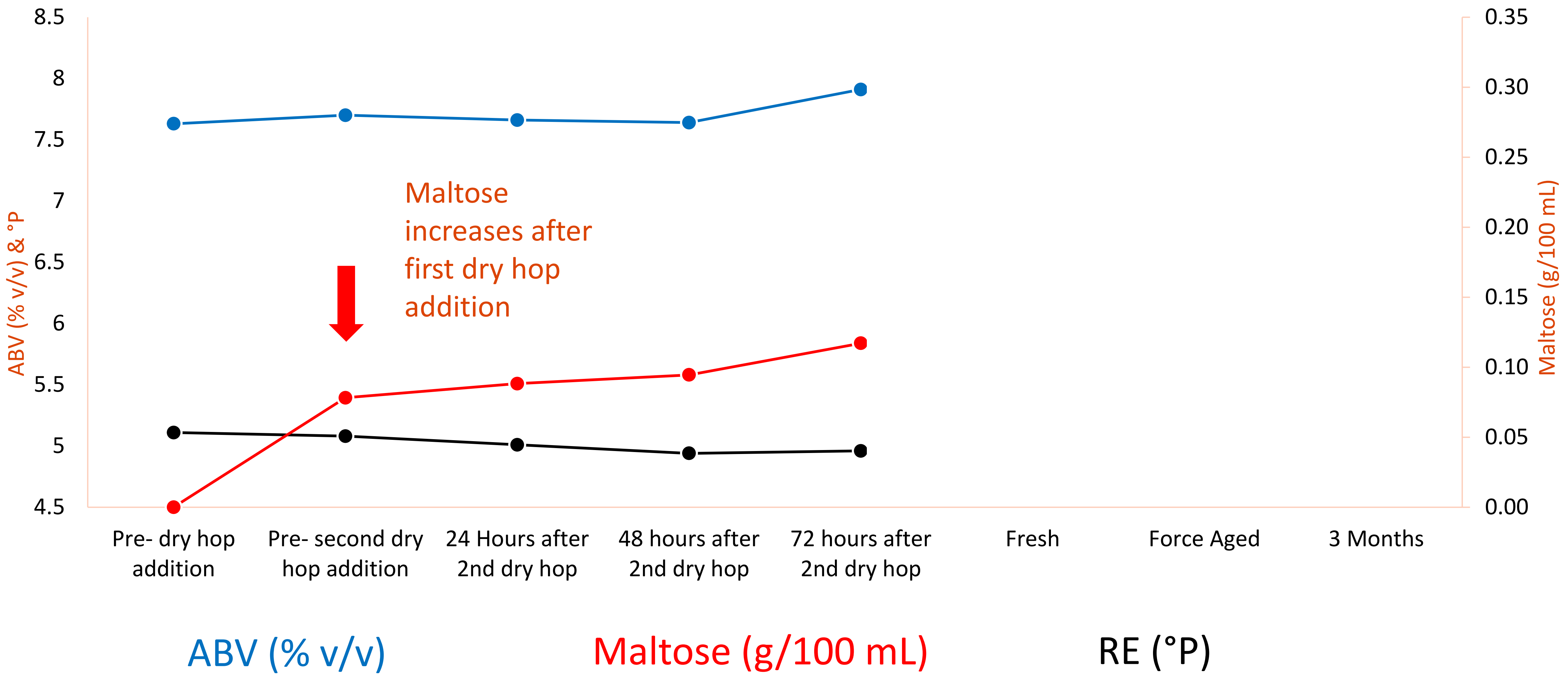
- Fresh
- Force aged (3 days @ 37° C)
- 3 months 25C (packaged 3 months earlier)

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

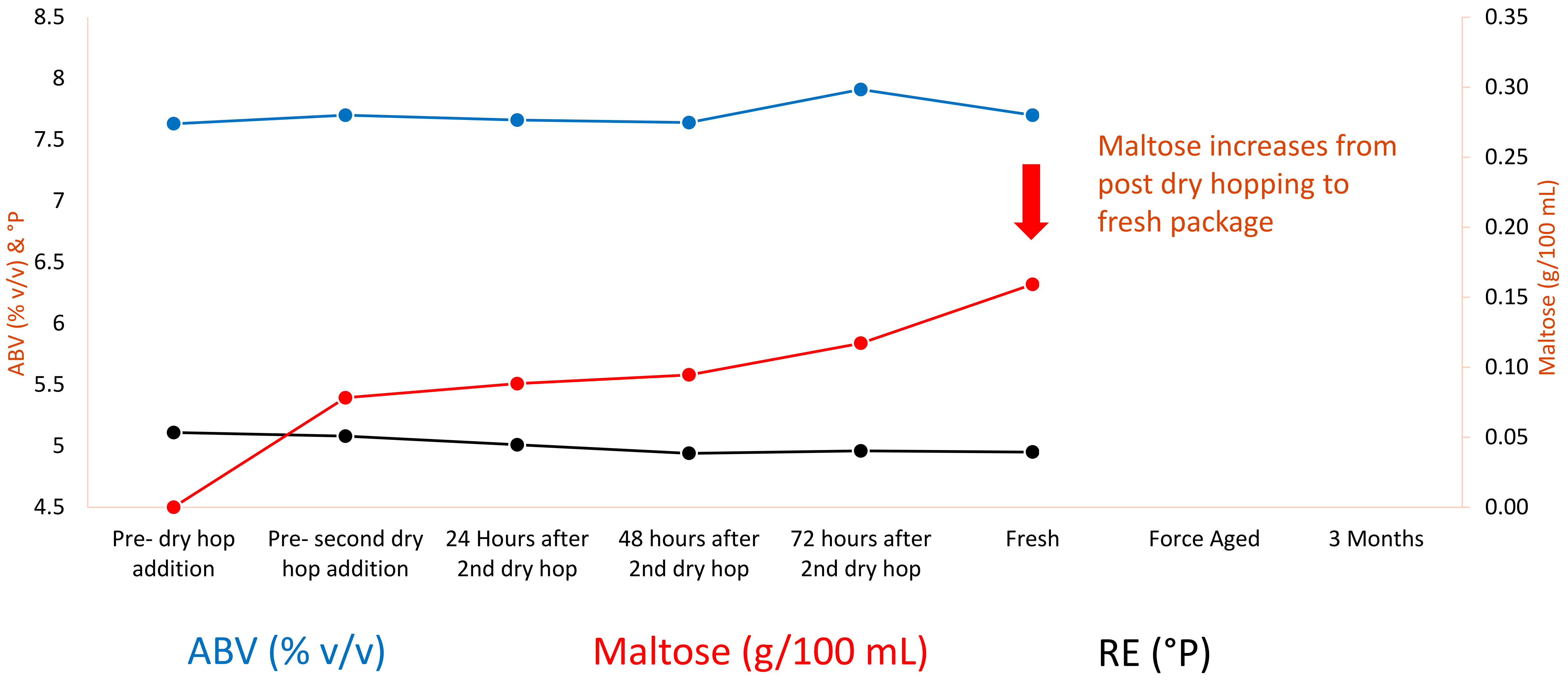
Results: before dry-hopping



Results: during dry-hopping

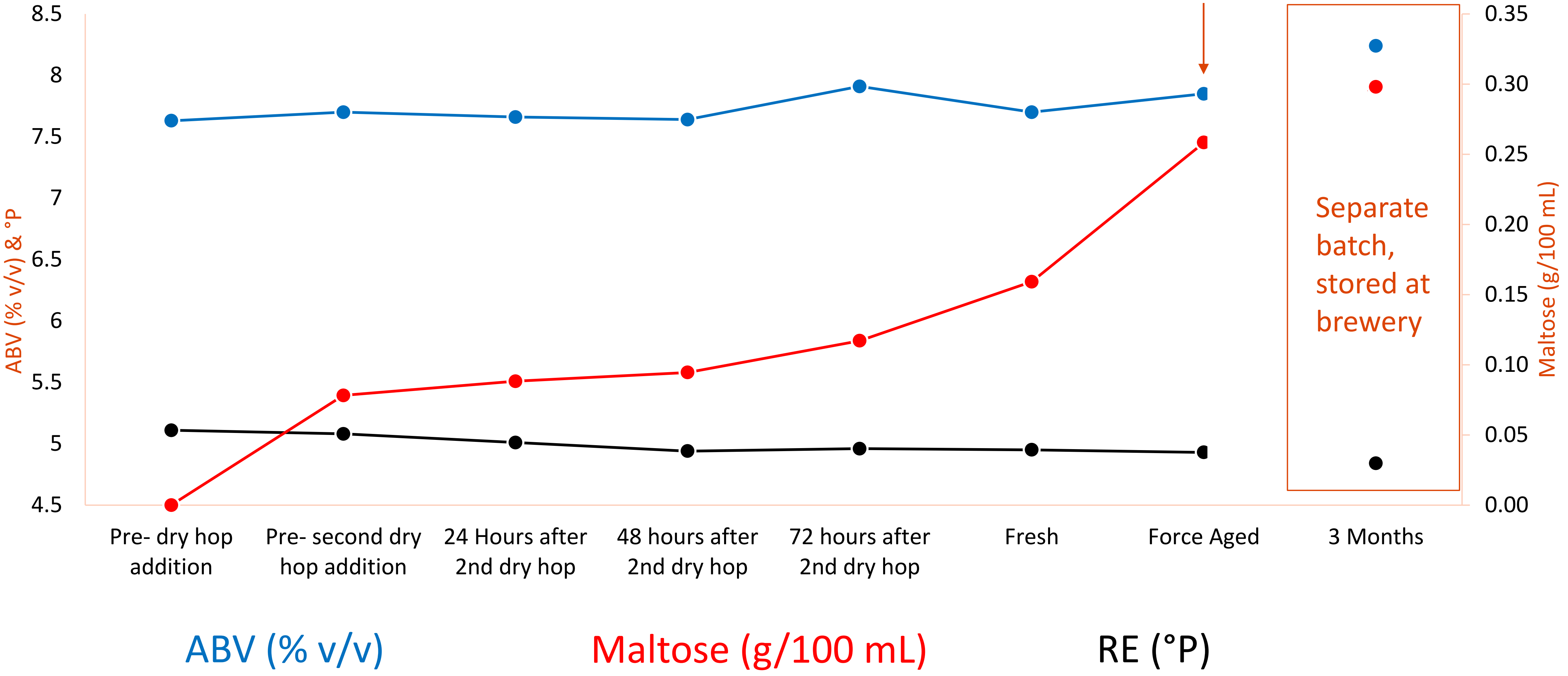


Results: fresh



Results: force aged 37°C

Increased maltose and ABV in forced aged (37C) for 3 days



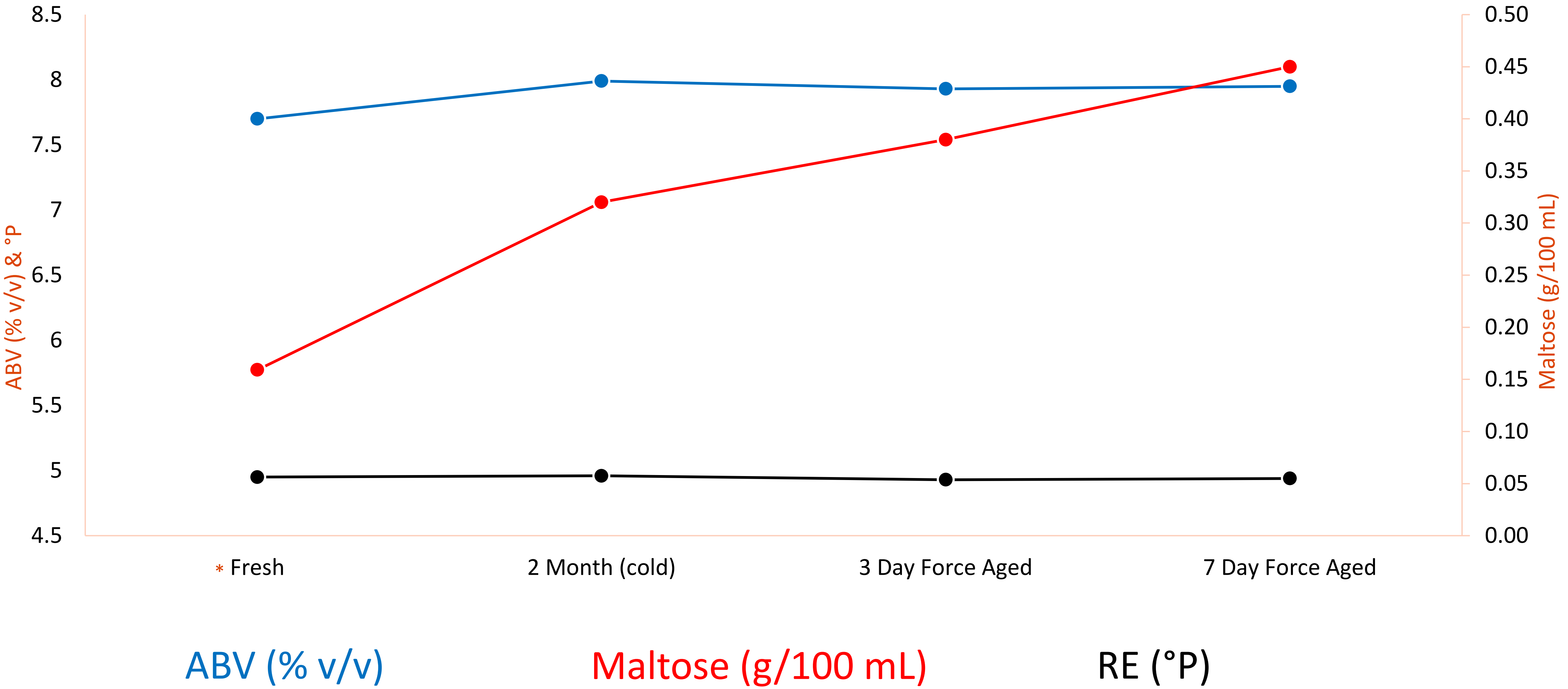
Continued aging study – 2 months later

- 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?

2 months 4°C	2 months 4°C + force aged x 3 days 37°C	2 months 4°C + force aged x 7 days 37°C

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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Dates	
	Online: May 14 - June 18, 2018
	Onsite: June 18 - 22, 2018

★ Introduction

Congratulations to the Pink Boots Society's 2018 winner of the Oregon State Beer Quality and Analysis series scholarship winner **Jocelyn Havel**! Learn more about Jocelyn and the Pink Boots Society. We look forward to welcoming Jocelyn and many other talented brewers and quality assurance specialists to Oregon in June. Please join us!

Thank you

Oregon State University