Dry-hopping Beer and Achieving Consistent Flavor

Shellhammer Laboratory
Department of Food Science and Technology
WHIRLPOOL VS. DRY-HOPPING: SENSORY ANALYSIS
Hop Aroma: Late vs. Dry Hopping

- Background
- Basics of Experimental Design and Setup
- Results
Background and literature review


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

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

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

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

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.

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 boil
  (* 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
SENSORY RESULTS
PCA biplot of Descriptive Data

- Control
- F2 (8.5%)
- F1 (79.5%)

- Descriptor
- Group 1
- Group 2
- Group 3

- Amarillo DH
- Chinook DH
- Citra DH
- Citra WP
- Galaxy DH
- Galaxy WP
- Mosaic DH
- Mosaic WP
- Nelson Sauvin DH
- Nelson Sauvin WP
- Simcoe DH
- Simcoe WP
- Saaz DH
- Saaz WP
- Nugget DH
- Nugget WP
- Hudlertau MF DH
- Hudlertau MF WP
- Huell Melon DH
- Huell Melon WP
- Cascade DH
- Cascade WP

- Sharp, 2016
PCA biplot of Descriptive Data

- Sharp, 2016

- **Group 1**
  - Amarillo DH
  - Mosaic DH
  - Saaz DH
  - Simcoe WP
  - F1 (79.5%)

- **Group 2**
  - Citra DH
  - Mosaic WP
  - Simcoe WP
  - F2 (8.5%)

- **Group 3**
  - Nelson Sauvin DH
  - Citra WP
  - OHAI

- **Descriptors**
  - Sharp, 2016
  - F1 (79.5%)
  - F2 (8.5%)
  - green/vegetal
  - herbal/spicey
  - pine/resinous
  - citrus
  - fruity
  - catty/sweaty
  - OHAI

- **Varieties**
  - Amarillo WP
  - Cascade WP
  - Chinook WP
  - Galaxy WP
  - Hallertau MF DH
  - Huell Melon WP
  - Kernza DH
  - Nugget DH
  - Nelson Sauvin DH
  - Saaz WP
  - Simcoe WP
  - Nugget WP
PCA biplot of Descriptive Data

Sharp, 2016
PCA biplot of Descriptive Data

- Group 3

Descriptors:
- Herbal/spicy
- Pine/resinous
- Tropical fruit
- Green/vegetal
- Floral
- Citrus
- Fruity
- Catty/sweaty

Sharp, 2016
PCA biplot of Descriptive Data

- F1 (79.5%)
- F2 (8.5%)
- Amarillo
- Simcoe
- Galaxy
- Neibson Sauvin
- Columbus
- Columbus
- Citra
- Mosaic
- Citra
- OHAI

Descriptors:
- 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
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

<table>
<thead>
<tr>
<th>Region</th>
<th>Farm (coded)</th>
<th>OSU Hop Oil (ml/100g)</th>
</tr>
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<tbody>
<tr>
<td>CAS_12_16</td>
<td>WA 2</td>
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<tr>
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13 Unique oil values
Dry hopped at 3.8g/L

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<tr>
<td>CAS_14_16</td>
<td>WA 2</td>
<td>2.6</td>
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</table>

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

- 0 g/hL
- 2 g/L (0.5 lb/Bbl)
- 3.8 g/L (1 lb/Bbl)
- 8 g/L (2 lb/Bbl)
- 16 g/L (4 lb/Bbl)
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
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
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

Evaluations using draft beer

- Minimized total package oxygen
- Great for sensory testing implementation
Sensory evaluation - descriptive analysis
### Sensory evaluation – descriptive analysis external controls

<table>
<thead>
<tr>
<th>Attributes</th>
<th>Base (No dry hop)</th>
<th>3.8 g/L</th>
<th>16 g/L</th>
<th>Ballast Point Grapefruit Sculpin</th>
<th>Hop Valley Citrus Mistress</th>
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<td>Overall Hop Aroma Intensity</td>
<td>0</td>
<td>8-9</td>
<td>14-15</td>
<td>14-15</td>
<td>7-8</td>
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<tr>
<td>Citrus</td>
<td>0</td>
<td>7-8</td>
<td>5-6</td>
<td>13-14</td>
<td>6-7</td>
</tr>
<tr>
<td>Herbal/Tea</td>
<td>0</td>
<td>5-6</td>
<td>12-13</td>
<td>1-2</td>
<td>6-7</td>
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</tbody>
</table>

- Panelists came to consensus for attributes on commercial and internally made samples
- References were served to panelists at each DA session
SENSORY RESULTS
Hop dosage rate – Overall Hop Aroma Intensity

![Graph showing the relationship between dry hop rate (g/L) and sensory score on a 15pt scale. The x-axis represents dry hop rate (g/L) ranging from 0 to 18, and the y-axis represents sensory score ranging from 0 to 14. The graph includes multiple data points indicated by triangles, suggesting a positive correlation between hop dosage rate and 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*
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)
Hop dose response – hoppy quality (citrus and herbal/tea)

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

*Letters represent Tukey’s HSD groupings*
• **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
Solid phase micro extraction GC/MS – Hop volatiles (In Beer)

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- Methyl Geranate
- Geraniol Acetate
- β-Caryophyllene
- α-Humulene
- β-Farnesene
- Gernyal Isobutyrate
Hop Compound Concentrations – Terpene Alcohols

*Average of 4 SPME-GC/MS instrumental runs

#Estimated values lower than LOQ
Hop Compound Concentrations – Terpene Alcohols

- Linalool (ppb) vs Dry hop rate (g/L)
  - Detection threshold 1 ppb
  - Estimated values lower than LOQ

- Nerol (ppb) vs Dry hop rate (g/L)
  - Detection threshold 4 ppb
  - Estimated values lower than LOQ

*Average of 4 SPME-GC/MS instrumental runs

Hop total oil + compositional analysis

Internal Standard
2-octanol

Target Hop Analytes
Linalool
Terpinen-4-ol
α-terpineol
Nerol
Phenyl Acetate
Geraniol
Geranial-citral
Methyl Geranate
Geraniol Acetate
β-Caryophellene
α-Humulene
β-Farnesene
Gernyal Isobutyrate
Hop Compound % Extraction – Terpene Alcohols

*Assuming 100% extraction based on hop oil hydrodistillation*
Dry hopping having other impacts on beer...

**RE-Bottle Carbonation?**

**pH-Flavor Stability?**

**Bitterness?**

* 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”
BITTERNESS OF DRY-HOPPED BEER

Thomas H. Shellhammer, PhD
Nor’Wester Professor of Fermentation Science
Oregon State University
How does the nonvolatile fraction influence dry-hopped beer quality?
How does the nonvolatile fraction influence dry-hopped beer quality?

- Cellulose/Lignin: 35%
- Protein: 15%
- Hop acids: 20%
- Essential oils: 3%
- Polyphenols/Tannins: 5%
- Carbohydrates: 4%
- Minerals: 8%
- Water: 10%
How does the nonvolatile fraction influence dry-hopped beer quality?

- Protein 15%
- Hop acids 20%
- Essential oils 3%
- Polyphenols/Tannins 5%
- Carbohydrates 4%
- Minerals 8%
- Water 10%
- Cellulose/Lignin 35%
Hop acids (up to ~20% of hops)

**Alpha Acids**

**Beta Acids**
Hop acids (up to ~20% of hops)

Alpha Acids \rightarrow \text{Isomerization} \rightarrow \text{Iso-Alpha Acids}
Hop acids (up to ~20% of hops)

Alpha Acids

Iso-Alpha Acids

Oxidation

Isomerization

Humulinones
(oxidized alpha acids)
Hop acids (up to ~20% of hops)

Beta Acids

Isomerization

Oxidation

X

Hulupones
(oxidized beta acids)
Hop acids – what you may find in beer

Alpha Acids

Humulinones (oxidized alpha acids)

Iso-Alpha Acids

Hulupones (oxidized beta acids)
Hop acids – what you may find in beer

**Alpha Acids**

**Humulinones**
(oxidized alpha acids)

**Iso-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

- ABV (%)
- RE (%)
- pH
- Alpha (mg/L)
- Iso (mg/L)
- Humulinones (mg/L)
- TPP (mg/L)
Bitterness comes from Isos & Humulinones
BU predicts bitterness
Bitterness comes from Isos & Humulinones
BU predicts bitterness
HOP CREEP
Typical fermentation, no dry-hopping
Dry-hopping can create “Hop Creep”

Dry hop addition

Days after start of fermentation

Density (g/m³)
Cascade hops have broad (low) enzyme activities

<table>
<thead>
<tr>
<th>Enzyme</th>
<th>Hops</th>
<th>Malt (130 dp)</th>
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<tbody>
<tr>
<td>α-amylase</td>
<td>0.35</td>
<td>198</td>
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<td>β-amylase</td>
<td>0.41</td>
<td>13</td>
</tr>
<tr>
<td>Amyloglucosidase</td>
<td>0.02</td>
<td>NA</td>
</tr>
<tr>
<td>Limit dextrinase</td>
<td>&lt;0.01</td>
<td>NA</td>
</tr>
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</table>
Hop enzymes stimulate “after-fermentation”
AKA – Hop Creep

Days

Real Extract (°P)

Beer + Hops
Beer
Beer + Yeast
Beer + Yeast + Hops

OREGON STATE UNIVERSITY
Hop Creep & Diacetyl issues

Diacetyl (ppb)

Density (g/m³)

Days after start of fermentation
Hop Creep & Diacetyl issues

Dry hop addition

Days after start of fermentation

Density (g/m³)

Diacetyl (ppb)

OREGON STATE UNIVERSITY
HOP ENZYMES PERSIST IN PACKAGED BEER
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

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

Finished beer samples

ABV (% v/v)  Maltose (g/100 mL)  RE (°P)
Results: before dry-hopping

<table>
<thead>
<tr>
<th>Time</th>
<th>ABV (% v/v)</th>
<th>Maltose (g/100 mL)</th>
<th>RE (°P)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre- dry hop addition</td>
<td>7.63%</td>
<td>0.00 g/100 mL</td>
<td>5.11 °P</td>
</tr>
<tr>
<td>Pre- second dry hop add.</td>
<td>5.11 °P</td>
<td>0.00 g/100 mL</td>
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</tr>
<tr>
<td>24 Hours after 2nd dry hop</td>
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<td></td>
</tr>
<tr>
<td>48 hours after 2nd dry hop</td>
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<td>72 hours after 2nd dry hop</td>
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<tr>
<td>Force aged</td>
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<td>3 Months</td>
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Results: during dry-hopping

Maltose increases after first dry hop addition

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

Maltose increases from post dry hoppung to fresh package
Results: force aged 37°C

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

Separate batch, stored at brewery
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
+ 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
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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OSU's leading program for professional brewers offers the science of beer to analyze beer and influence quality control in a production brewing setting.

★ 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