
Harvest Maturity of Cascade and Willamette Hops



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“Maturity” not “Age”



AGE

IS A HIGH PRICE TO PAY FOR MATURITY

Hop maturity and its impact on hop oil composition

- Hops -Overview
 - Chemical composition
- Harvest Maturity
 - Design
 - Chemical
 - Sensory
- Conclusions
 - Practical considerations



Photo by: Jim Solberg, Indie Hops

Harvest Maturity of Cascade and Willamette Hops

- How does harvest timing affect the chemistry of Cascade and Willamette hops?
 - Previous work:
 - Increased essential oil content over time.¹
 - Compositional changes throughout maturity.²
 - Varietal constancy under various environmental factors.³

1. Murphey, J.; Probasco, G. The Development of Brewing Quality Characteristics in Hops During Maturation. *MBAA TQ* 1996, 33, 149-159.

2. Bailey, B.; Schonberger, C.; Drexler, G.; Gahr, A.; Newman, R. The Influence of Hop Harvest Date on Hop Aroma in Dry-Hopped Beers. *MBAA TQ* 2009.

3. Likens, S. T.; Nickerson, G. B. Identification of hop varieties by gas chromatographic analysis of their essential oils. Constancy of oil composition under various environmental influences. *Journal of Agricultural and Food Chemistry* 1967, 15, 525-530.

Chemical Composition of Hops

| Principle Components | Concentration (%w/w) |
|-------------------------|----------------------|
| Cellulose-lignins | 40.0 - 50.0 |
| Proteins | 15.0 |
| Alpha acids | 2.0 - 17.0 |
| Beta acids | 2.0 - 10.0 |
| Water | 8.0 - 12.0 |
| Minerals | 8.0 |
| Polyphenols and tannins | 3.0 - 6.0 |
| Lipids and fatty acids | 1.0 - 5.0 |
| Hop oil | 0.5 - 3.0 |
| Monosaccharides | 2.0 |
| Pectins | 2.0 |
| Amino acids | 0.1 |

European Brewery Convention *Hops and Hop Products, Manual of Good Practice*; Getränke - Fachverlag Hans Carl: Nurnberg, Germany, 1997.



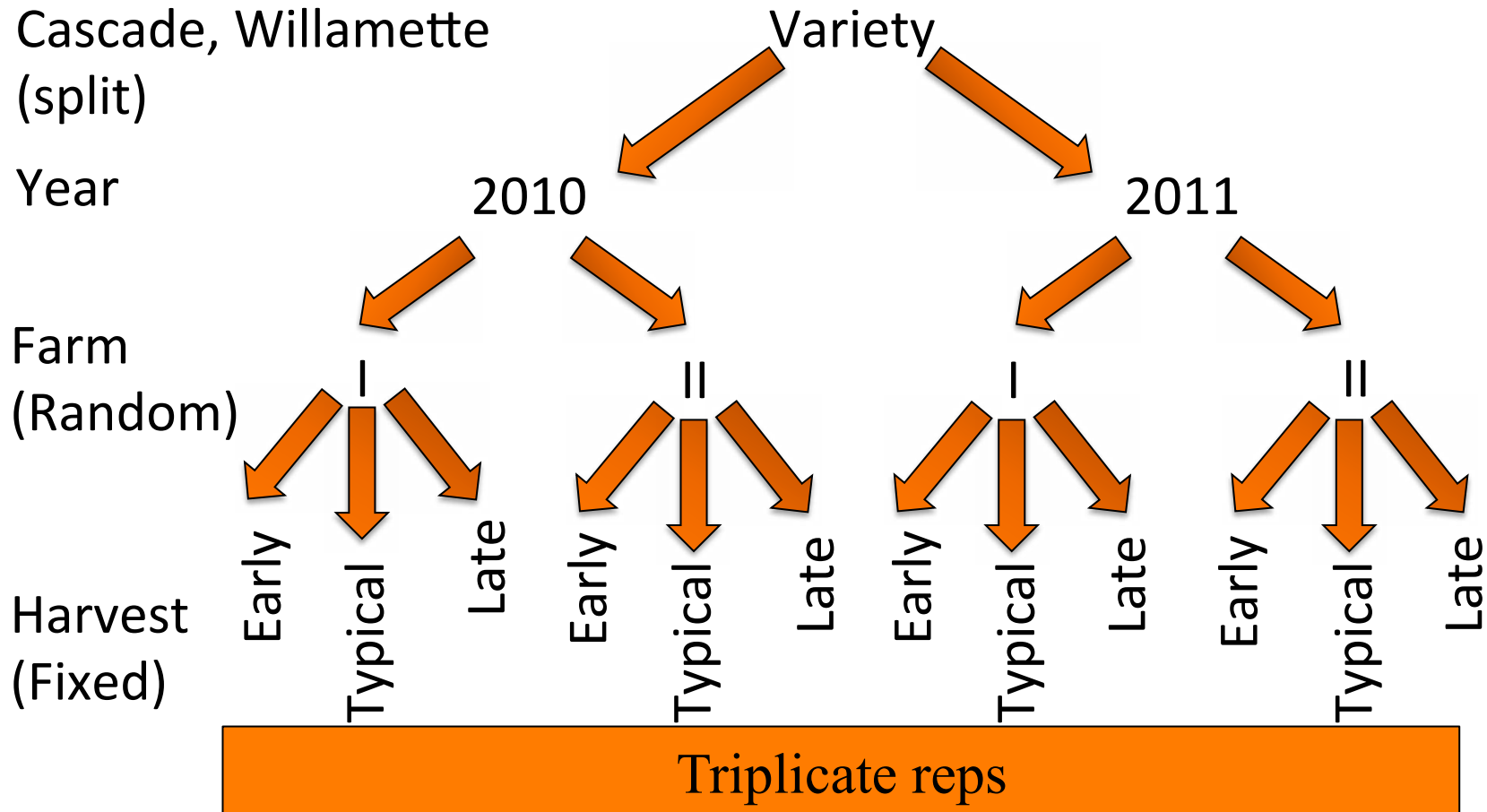
Kishimoto, T.; Wanikawa, A.; Kono, K.; Shibata, K. Comparison of the Odor-Active Compounds in Unhopped Beer and Beers Hopped with Different Hop Varieties. *Journal of Agricultural and Food Chemistry* 2006, 54, 8855-8861.



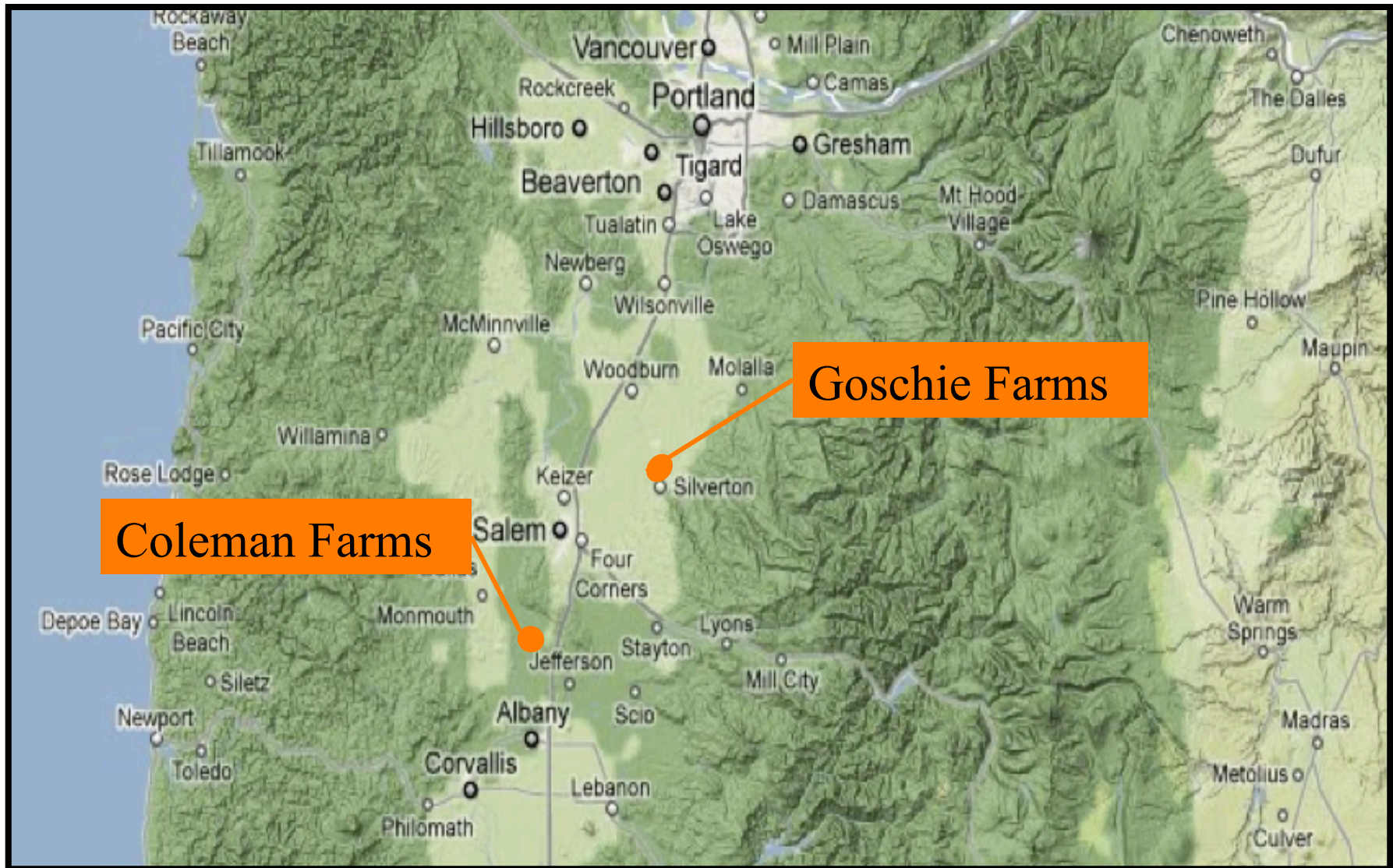
SEM Image of Lupulin glands

Forster, A. The quality chain from hops to hop products. In *48th IHGC Congress, Canterbury*, Barth-Haas Research & Publications (<http://www.barthhaas.com>); 2001.

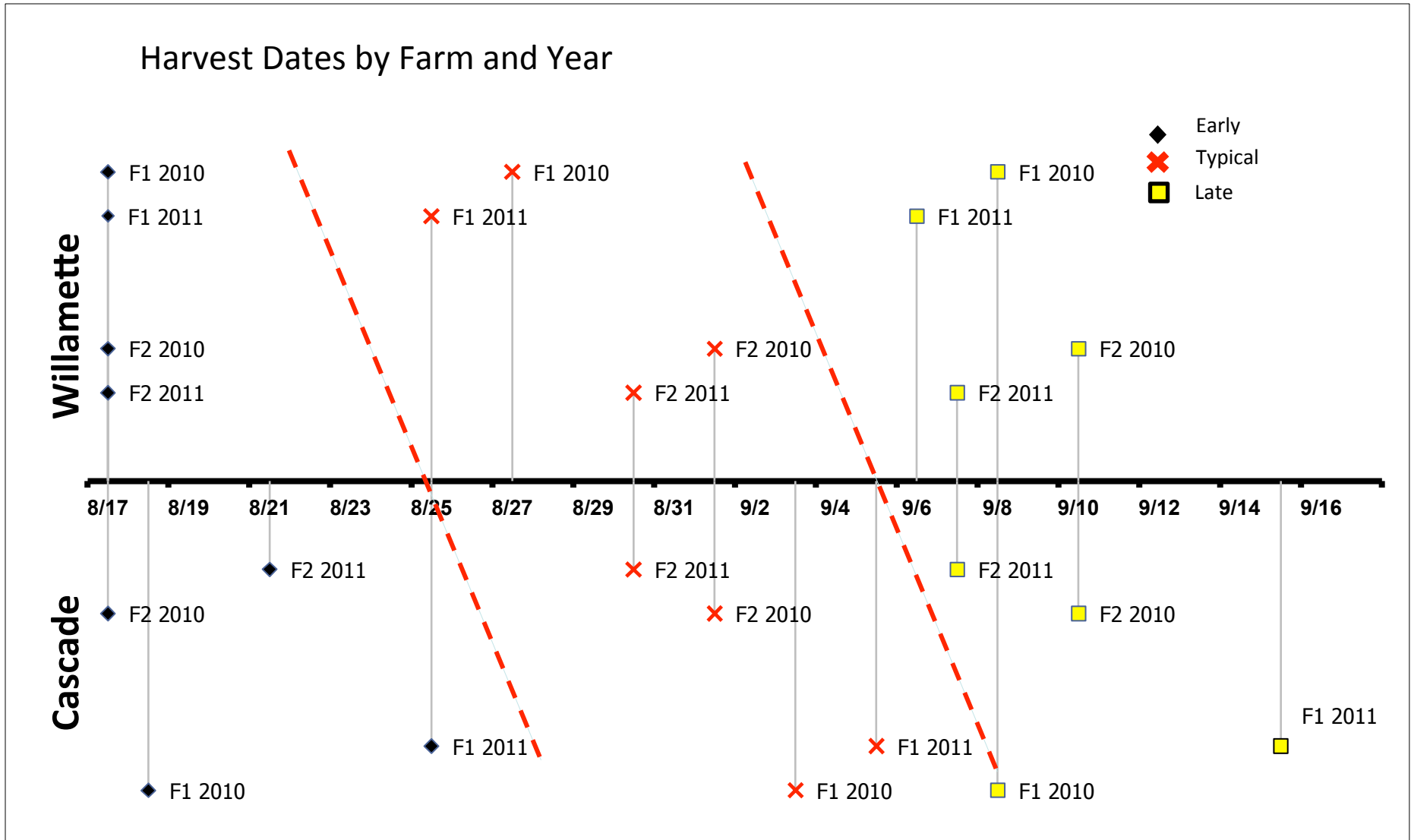
Study Design- Mixed Model



Participating Farms



Harvest Timeline



Experimental

- Hop Acids
 - Composition
 - Hop Storage Index
- Essential Oil
 - Total
 - Composition
- Moisture
 - Post kilning
- Sensory – 2010 Cascade
 - Difference
 - Acceptance
 - Preliminary Descriptive



Physical Examination



Experimental

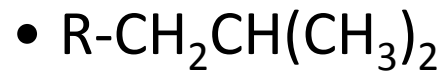
- **Hop Acids**
 - **Composition**
 - **Hop Storage Index**
- Essential Oil
 - Total
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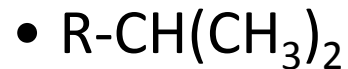
Alpha Acids and Hop Storage Index

- Bitterness precursors

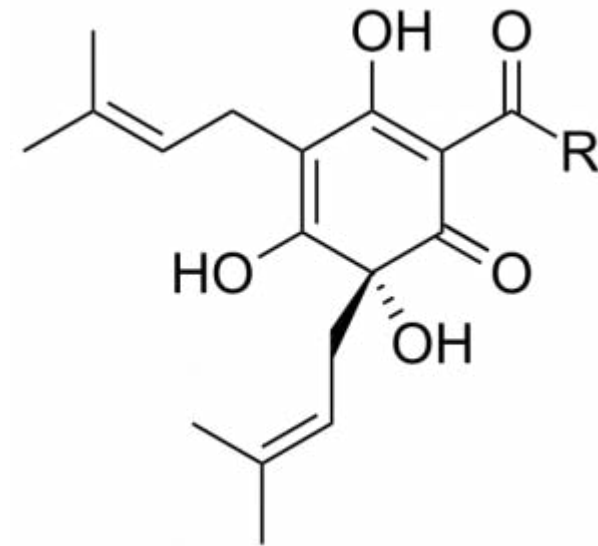
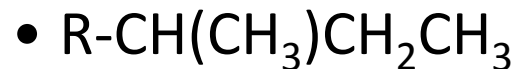
- Humulone



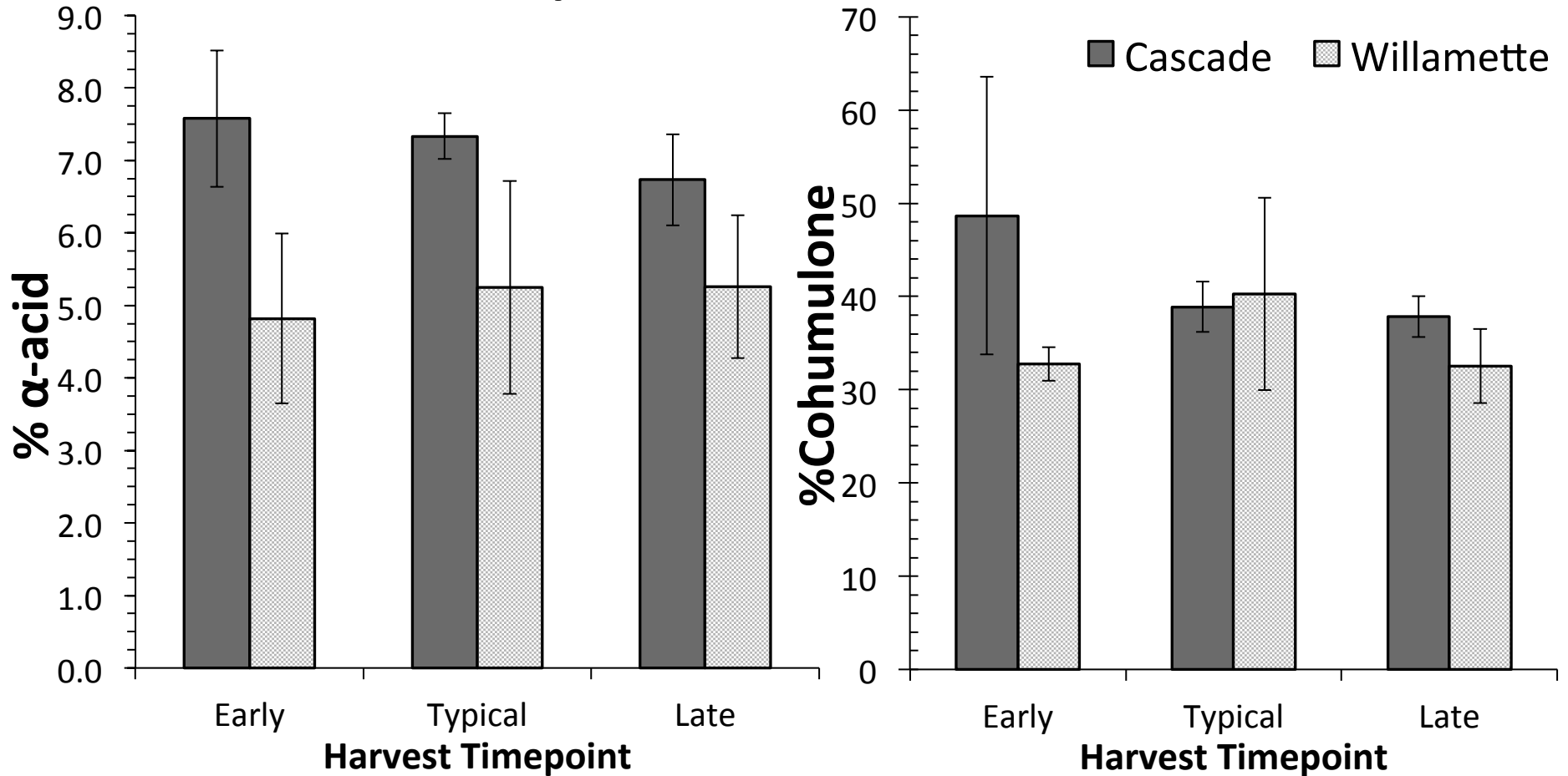
- Cohumulone



- Adhumulone



Alpha Acids (p-value > 0.05)



Hop Acids - UV absorbance

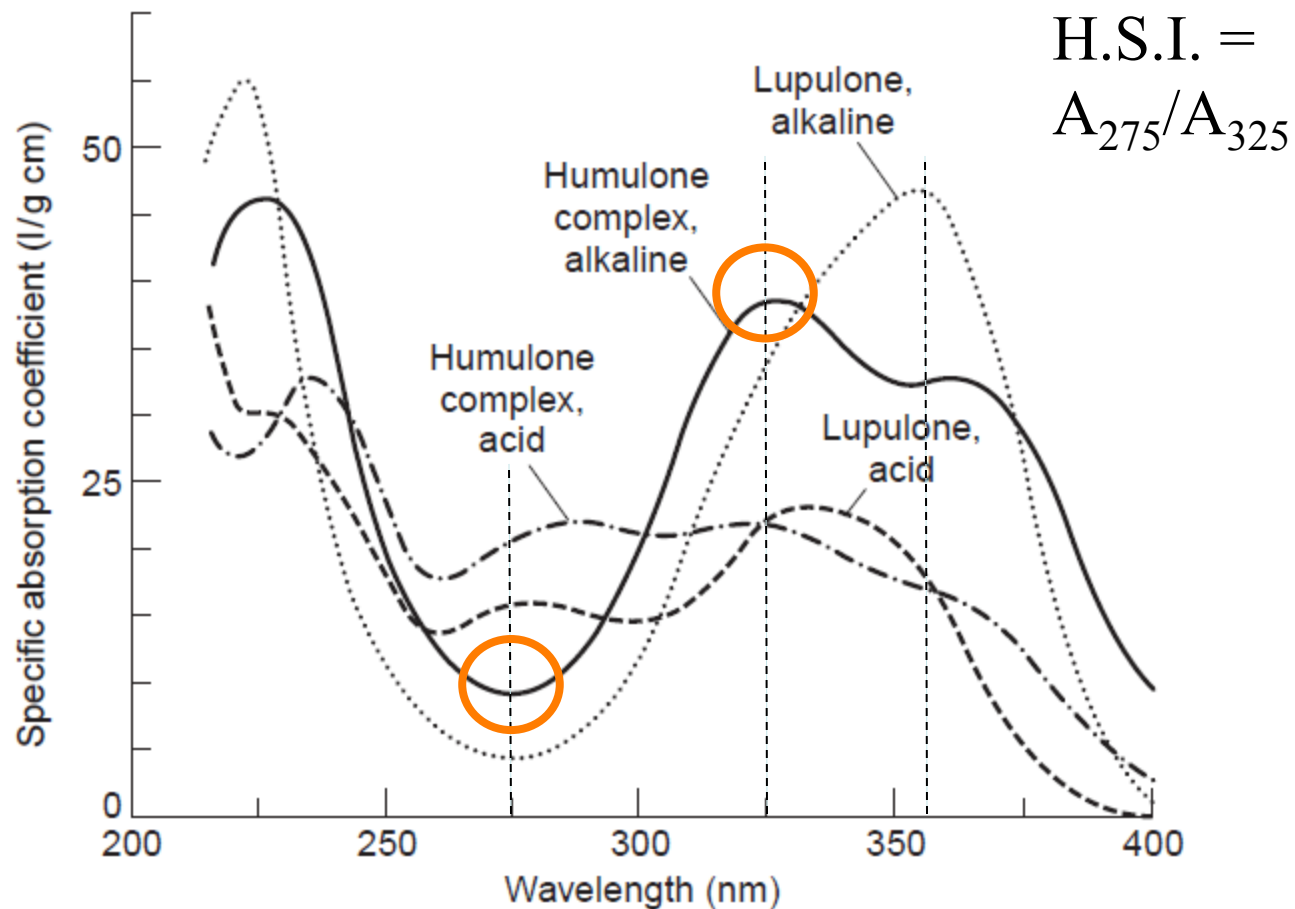
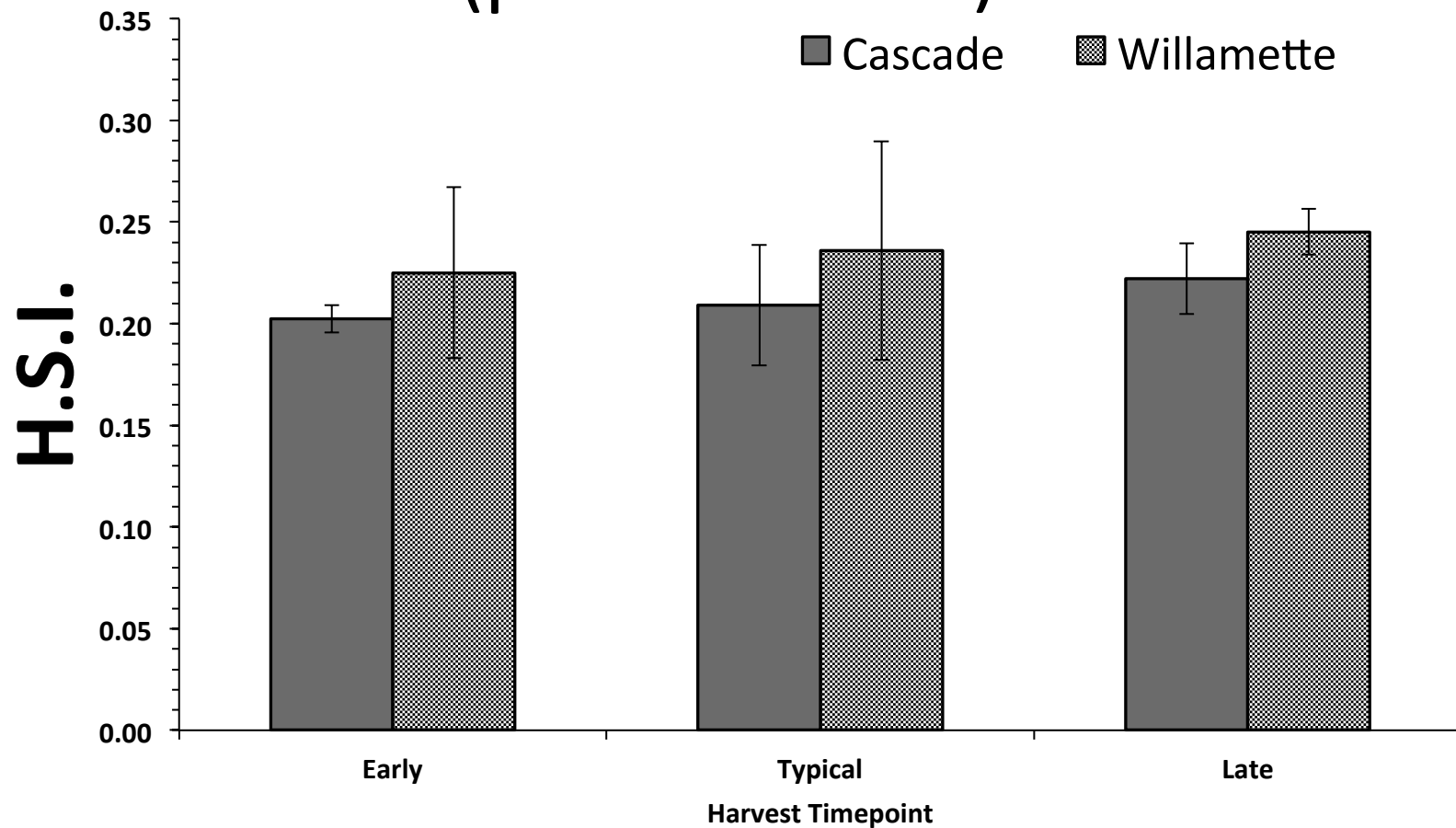


Fig. 8.6 Absorption spectra of lupulone and humulone complex in acidic (0.002N) and alkaline (0.002N) methanol (Alderton *et al.*, 1954). Copyright (1954) American Chemical Society.

Hop Storage Index (p-value > 0.05)

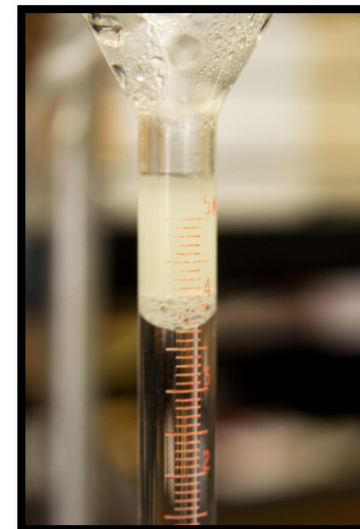
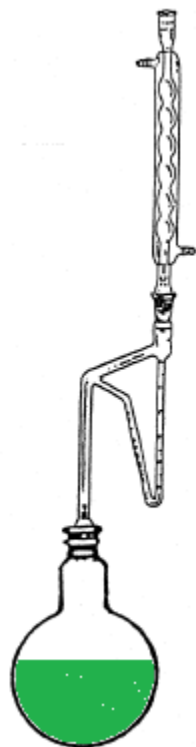


Experimental

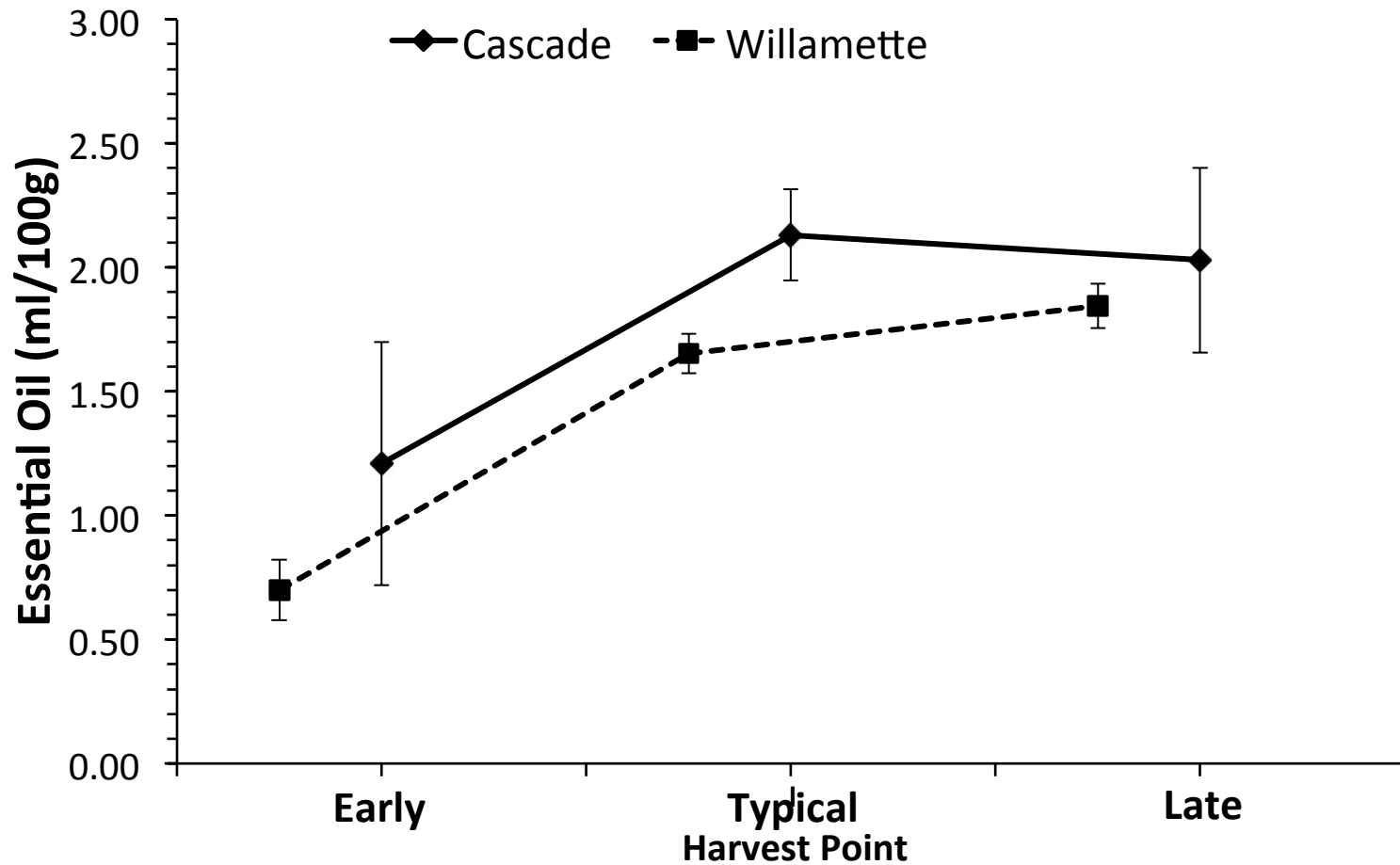
- Hop Acids
 - Composition
 - Hop Storage Index
- **Essential Oil**
 - **Total**
 - **Composition**
- Moisture
 - Post kilning
- Sensory
 - Difference
 - Acceptance
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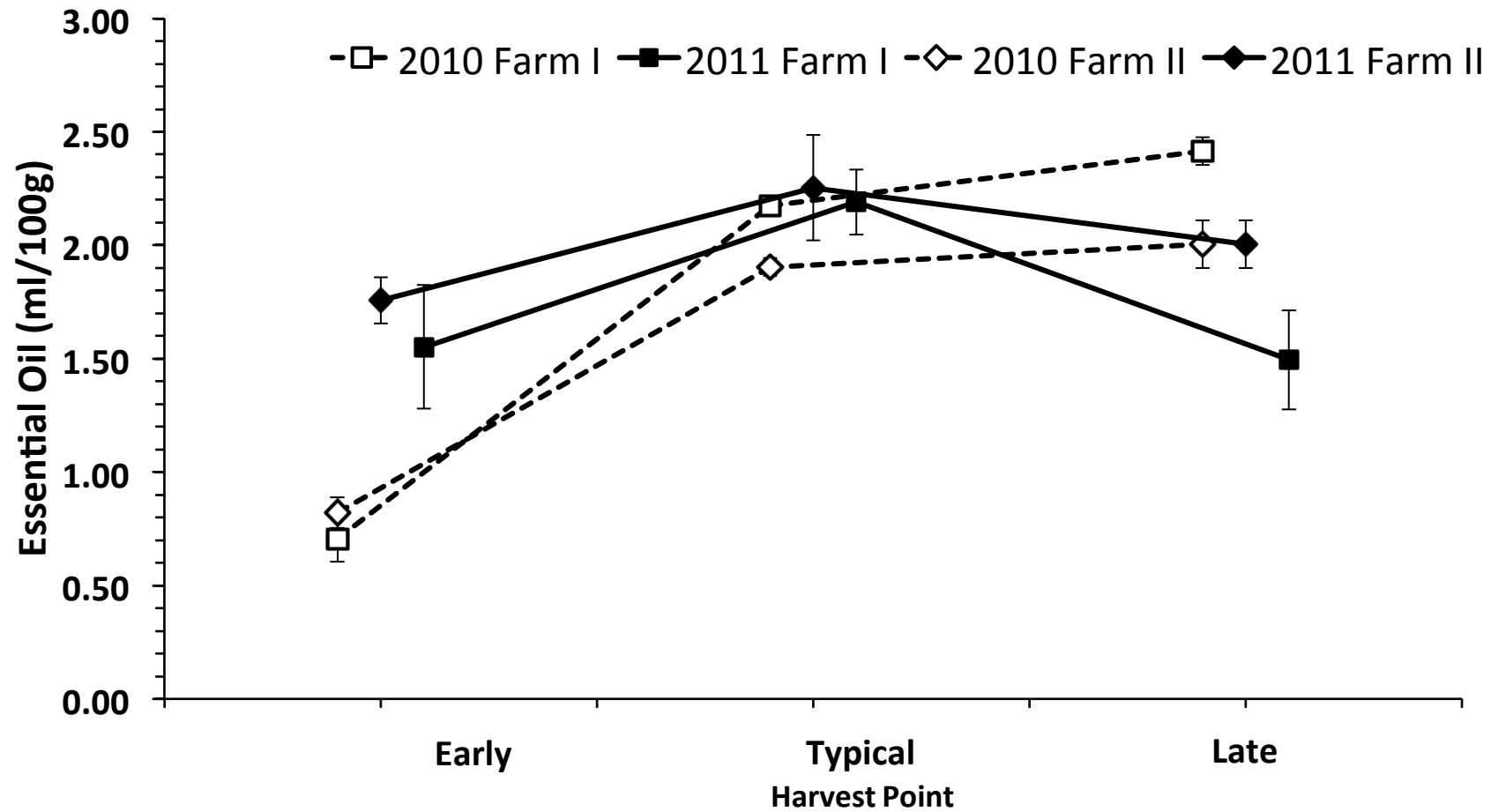
Essential Oil



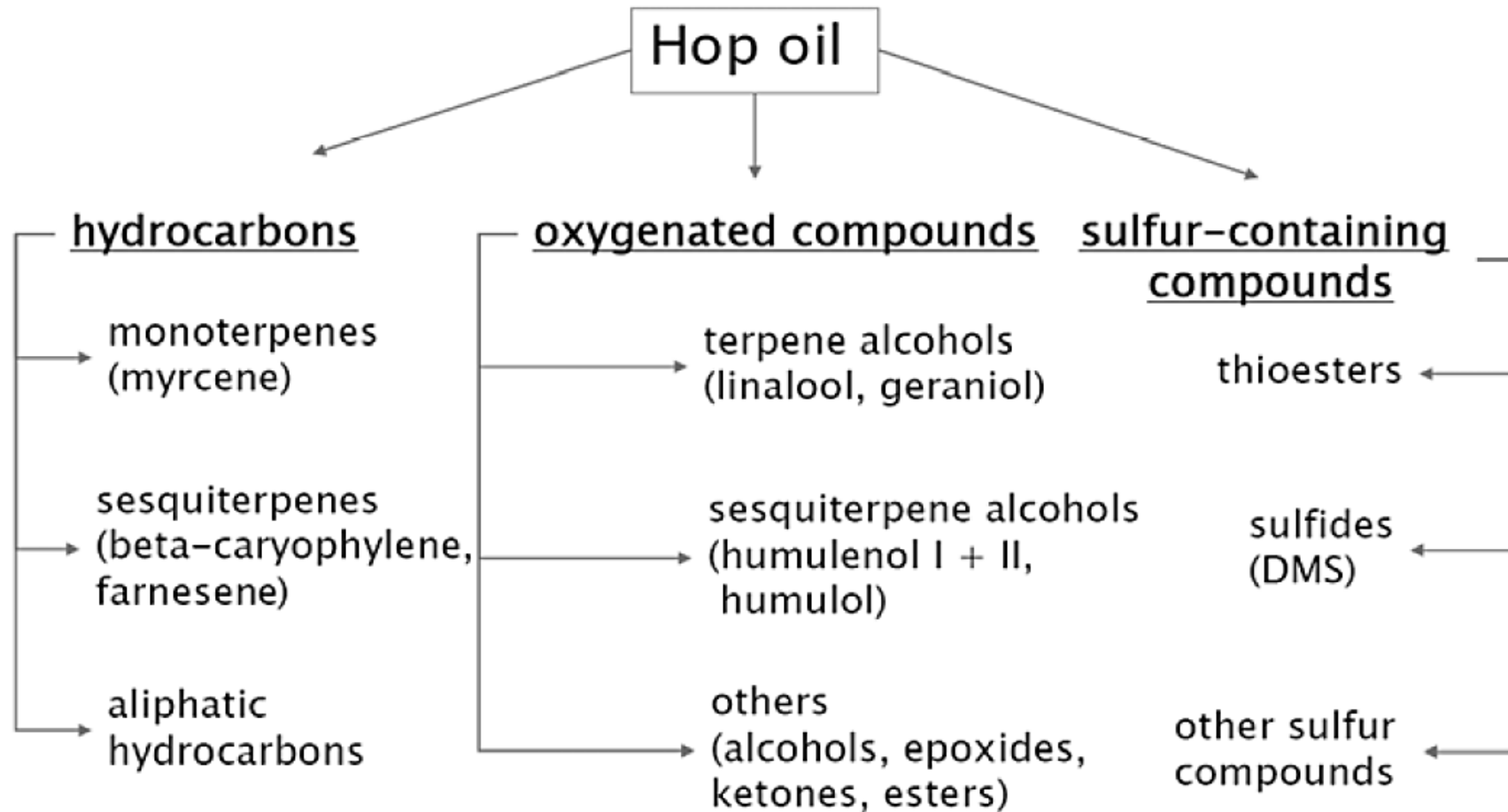
Total Essential Oil ($p < 0.05$)



Cascade Essential Oil

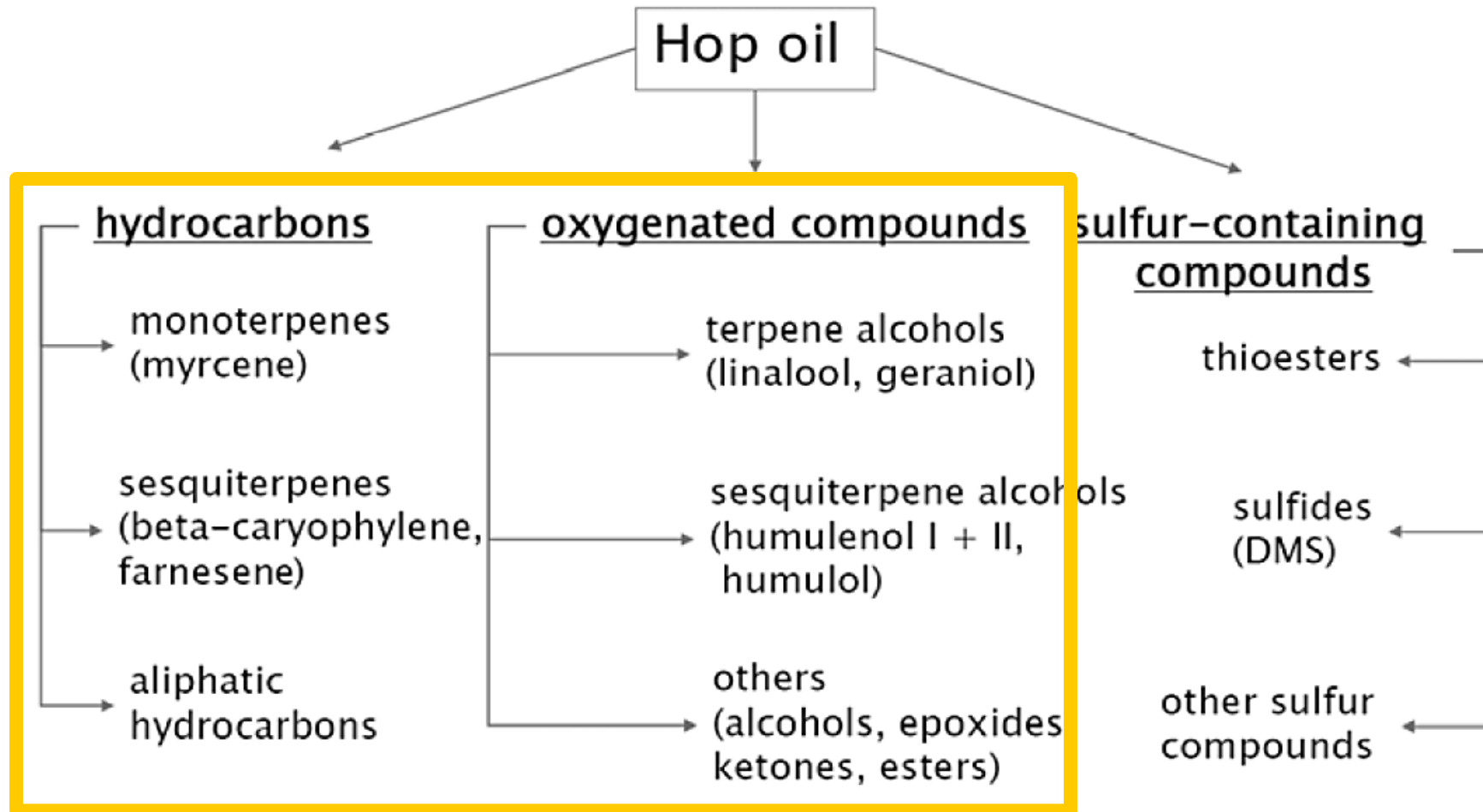


Hop Oil Composition



Schönberger, C.; Kostecky, T. 125th Anniversary Review: The Role of Hops in Brewing. *J. Inst. Brew* **2011**, *117*, 259–267.

Hop Oil Composition



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Hop Oil Compounds of Interest

| Compound Name | Classification | Description |
|-----------------------|-----------------------------------|---|
| α -pinene | Hydrocarbon, Monoterpene | Pine |
| β -pinene | Hydrocarbon, Monoterpene | Coniferous Pine, woody |
| β -Myrcene | Hydrocarbon, Monoterpene | Green, slightly metallic |
| Limonene | Hydrocarbon, Monoterpene | Citric, Orange |
| ρ -cymene | Hydrocarbon, Monoterpene like | Orange wood spice |
| Caryophyllene | Hydrocarbon, Sesquiterpene | Woody, Carrot |
| E, β -Farnesene | Hydrocarbon, Sesquiterpene | Green, woody, weedy, herbal, pine and gin |
| Humulene | Hydrocarbon, Sesquiterpene | Woody |
| Methyl heptanoate | Oxygenated, Ester | Sweet, fruity, peach, apricot, green, berry |
| Geraniol | Oxygenated, Monoterpene Alcohol | Sweet floral, perfumey |
| Linalool | Oxygenated, Monoterpene Alcohol | Floral, Orange |
| Citronellol | Oxygenated, Monoterpene | Floral, Rose Citrus |
| Farnesol | Oxygenated, Sesquiterpene Alcohol | Spicy |
| Citral | Oxygenated, Aldehyde | Sweet Citrus |
| Geranyl Acetate | Oxygenated, Monoterpene or ester | Floral, Sweet Citrus |
| Humulene Epoxide | Oxygenated, Epoxide | Spicy |
| Epoxide 2 | Oxygenated, Epoxide | Spicy |

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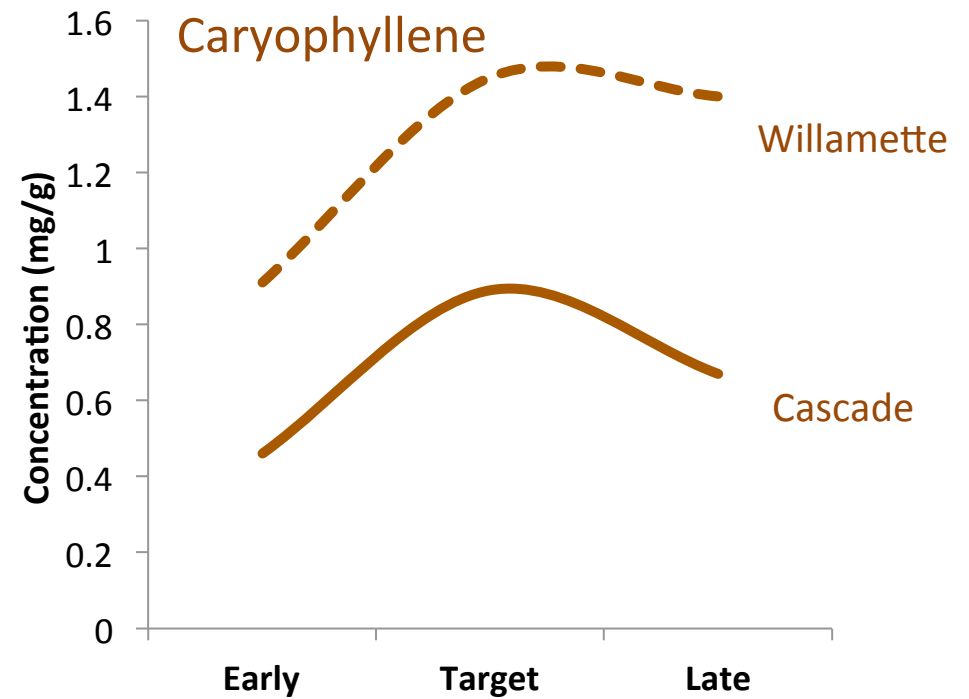
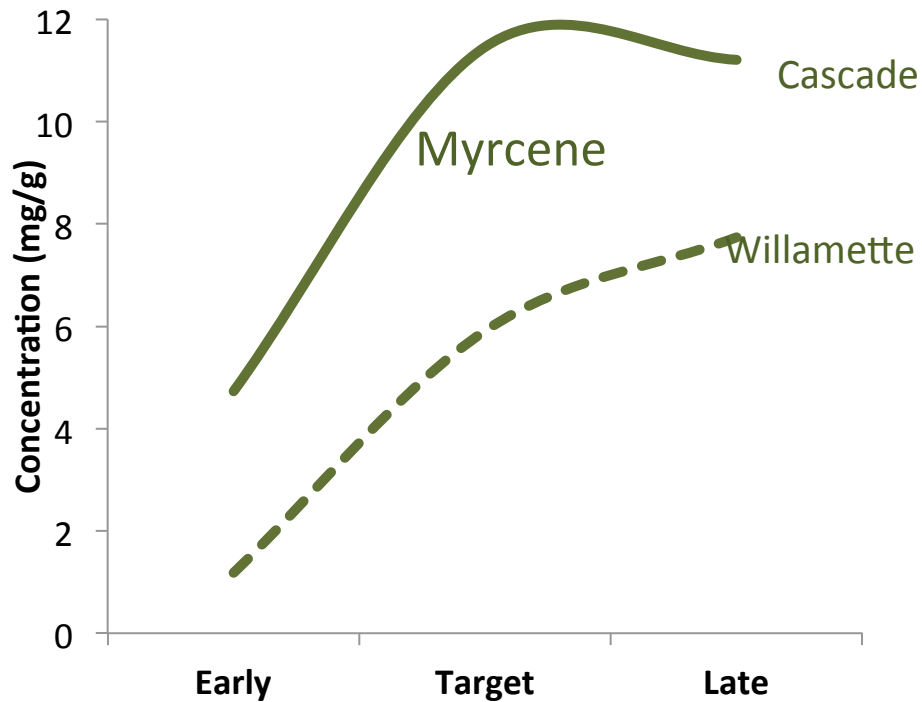
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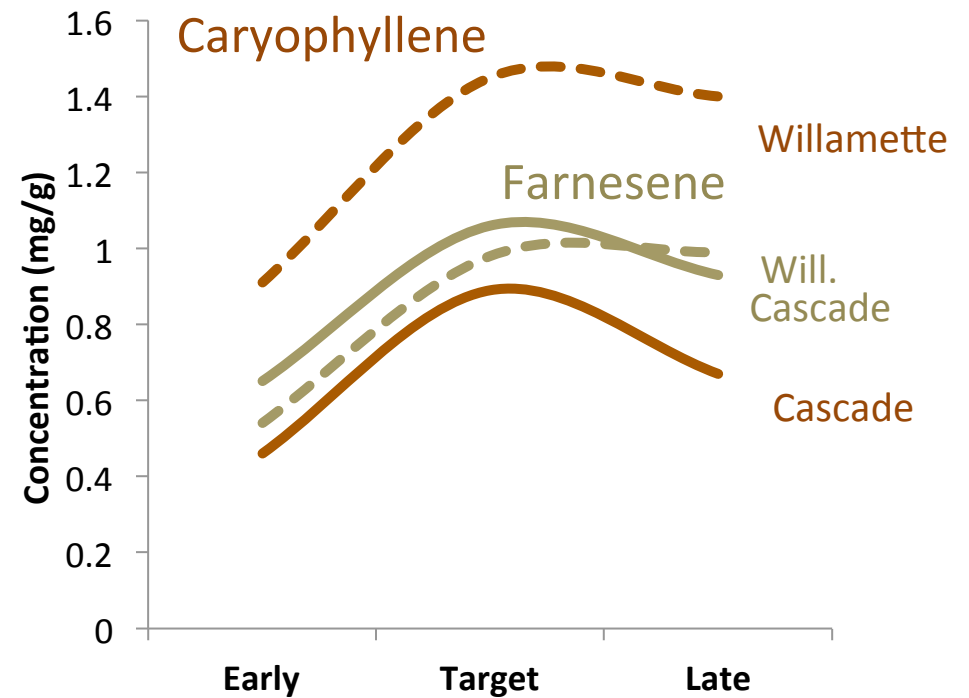
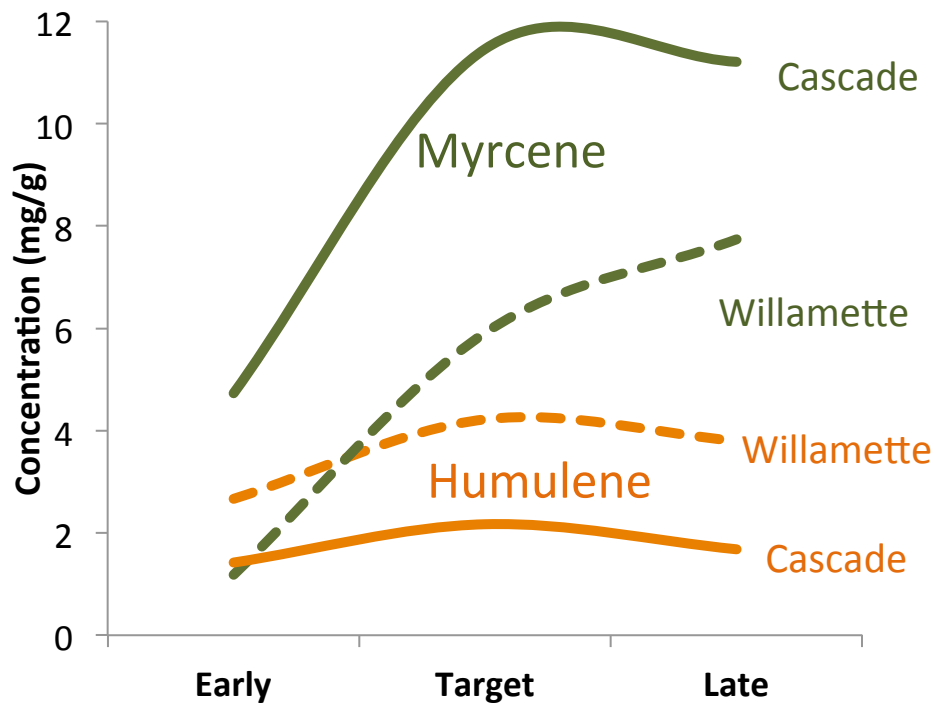
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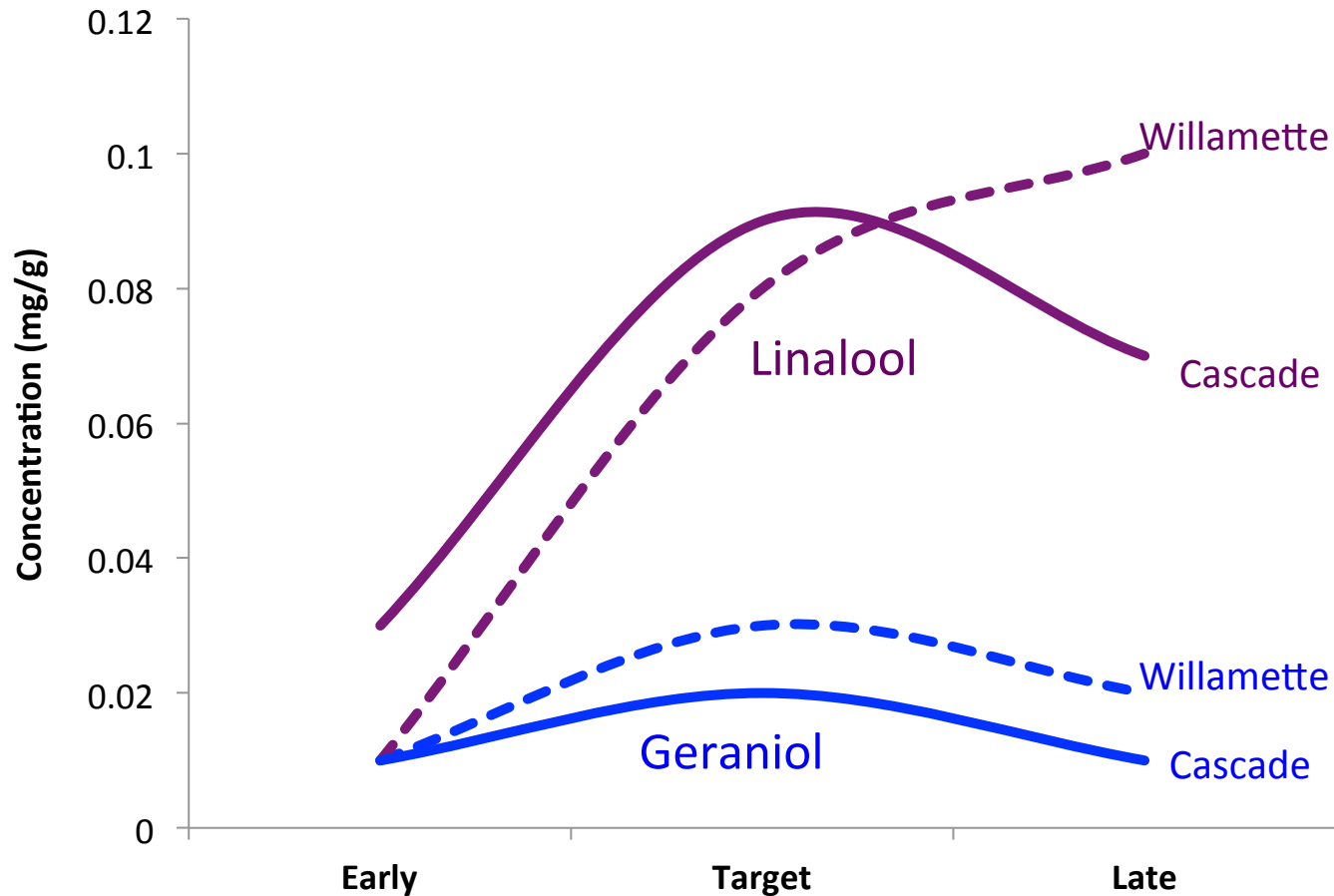
Changes in specific hop oil constituents



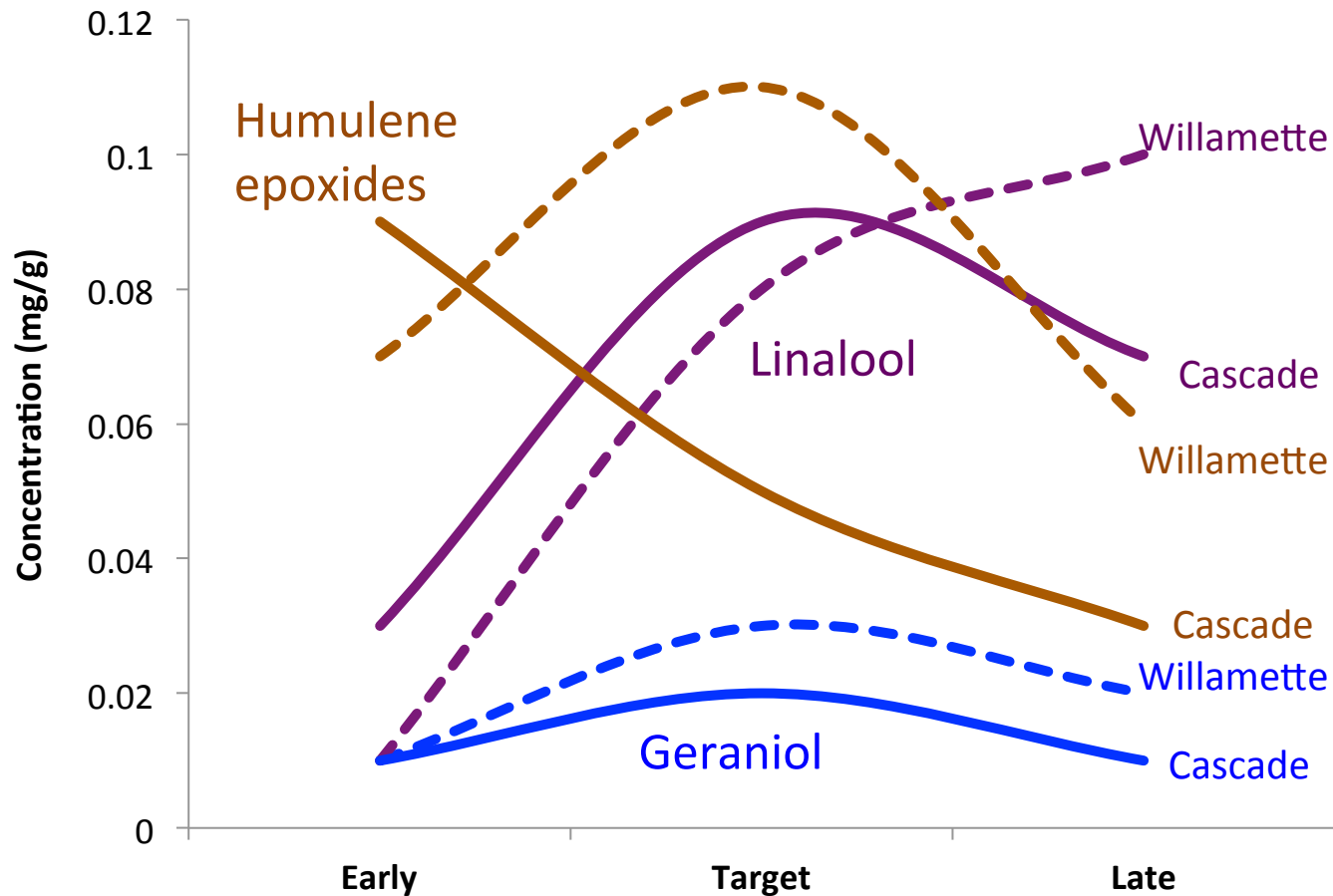
Changes in specific hop oil constituents



Changes in specific hop oil constituents



Changes in specific hop oil constituents



Chemical composition of hop essential oil

| Peak No. | Compound name | 1D-RT, 2-D RT [s] | Calculated RI | Literature RI | New |
|----------|---|-------------------|---------------|---------------|-----|
| 1 | Butanoic acid | 145, 2.00 | 845 | 820, H | no |
| 2 | Butanoic acid, 3-methyl- | 195, 2.57 | 876 | 877 | no |
| 3 | 1-Nonene | 225, 1.88 | 894 | 889, H | yes |
| 4 | Nonane | 235, 1.80 | | 900, H | yes |
| 5 | Pentanoic acid | 245, 2.77 | 907 | 911, H | no |
| 6 | 1,3-Nonadiene, (E)- | 265, 2.28 | 921 | 930 | yes |
| 7 | Methyl hexanoate | 265, 2.78 | 921 | 923 | no |
| 8 | 1 <i>F</i> - α -Pinene | 275, 2.37 | 929 | 934 | no |
| 9 | Camphene | 295, 2.51 | 943 | 953 | no |
| 10 | Benzaldehyde | 305, 4.61 | 950 | 962 | no |
| 11 | (1 <i>S</i>)- β -Pinene | 335, 2.75 | 971 | 980 | no |
| 12 | 1-Octen-3-ol | 345, 2.83 | 979 | 980 | no |
| 13 | 5-Hepten-2-one, 6-methyl- | 355, 3.47 | 986 | 987 | no |
| 14 | β -Myrcene | 365, 2.83 | 993 | 991 | no |
| 15 | Decane | 375, 2.05 | | 1000, H | yes |
| 16 | Hexanoic acid | 385, 3.17 | 1007 | 1019, H | no |
| 17 | β -Phellandrene | 405, 3.03 | 1020 | 1031 | no |
| 18 | D-Limonene | 415, 2.87 | 1027 | 1031 | yes |
| 19 | Methyl heptanoate | 415, 3.06 | 1027 | H | no |
| 20 | Benzyl alcohol | 415, 5.13 | 1027 | 1032 | no |
| 21 | (Z)- β -Ocimene | 425, 2.92 | 1033 | 1037 | no |
| 22 | Benzeneacetaldehyde | 425, 5.42 | 1033 | 1043 | yes |
| 23 | (E)- β -Ocimene | 445, 2.92 | 1047 | 1048 | no |
| 24 | γ -Terpinene | 455, 3.13 | 1053 | 1062 | no |
| 25 | 2-Nonanol | 475, 2.87 | 1067 | 1087 | no |
| 26 | 1-Octanol | 485, 3.10 | 1073 | 1075 | no |
| 27 | 1-Undecene | 515, 2.35 | 1093 | 1092, H | yes |
| 28 | 2-Nonanone | 515, 3.37 | 1093 | 1093, H | no |
| 29 | Undecane | 525, 2.23 | | 1100, H | no |
| 30 | Heptanoic acid | 535, 3.42 | 1106 | 1085, H | no |
| 31 | Phenylethyl alcohol | 545, 5.40 | 1112 | 1118 | yes |
| 32 | Methyl octanoate | 565, 3.33 | 1125 | 1124, H | no |
| 33 | L-Pinocarveol | 575, 4.15 | 1131 | 1139 | no |
| 34 | (+)-Camphor | 585, 4.67 | 1138 | 1147 | yes |
| 35 | Sabina ketone | 605, 5.12 | 1150 | 1156 | yes |
| 36 | Borneol | 625, 4.13 | 1162 | 1165 | no |
| 37 | 1-Nonanol | 645, 3.25 | 1175 | 1175 | yes |
| 38 | 4-Terpineol | 645, 3.90 | 1175 | 1182 | no |
| 39 | <i>p</i> -Cymen-8-ol | 655, 4.83 | 1181 | 1186 | yes |
| 40 | Cryptone | 655, 5.08 | 1181 | 1188 | yes |
| 41 | (+)- α -Terpineol (<i>p</i> -menth-1-en-8-ol) | 665, 4.08 | 1188 | 1198 | no |
| 42 | Myrtenal | 665, 5.05 | 1188 | 1193 | yes |
| 43 | Methyl salicylate | 665, 5.20 | 1188 | 1190 | no |
| 44 | 1-Dodecene | 675, 2.45 | 1194 | 1192, H | yes |
| 45 | Myrtenol | 675, 4.38 | 1194 | 1194 | yes |
| 46 | Dodecane | 685, 2.32 | | 1200, H | no |
| 47 | Octanoic Acid | 685, 3.51 | 1200 | 1179, H | no |
| 48 | Acetic acid, octyl ester | 705, 3.27 | 1212 | 1200 | no |
| 49 | β -Cycloctral | 705, 4.72 | 1212 | 1219 | yes |
| 50 | <i>trans</i> -Carveol | 715, 4.32 | 1219 | 1217 | yes |
| 51 | Methyl nonanoate | 725, 3.44 | 1225 | 1224 | no |
| 52 | Nerol | 735, 3.87 | 1231 | 1242 | no |
| 53 | Hexyl 2-methylbutyrate | 745, 3.18 | 1238 | 1239 | yes |
| 54 | Neral | 745, 4.33 | 1238 | 1240 | no |
| 55 | <i>S</i> -Carvone | 745, 4.98 | 1238 | 1242 | no |
| 56 | Geranio | 775, 4.07 | 1256 | 1255 | no |
| 57 | Geranial | 795, 4.40 | 1269 | 1268 | no |
| 58 | Nonanoic acid | 825, 3.60 | 1288 | 1276, H | no |
| 59 | 1-Tridecene | 835, 2.53 | 1294 | 1292, H | yes |
| 60 | Tridecane | 845, 2.40 | 1290 | 1290, H | yes |

| Peak No. | Compound name | 1D-RT, 2-D RT [s] | Calculated RI | Literature RI | New |
|----------|-------------------------------------|-------------------|---------------|---------------|-----|
| 61 | 2-Undecanone | 845, 3.42 | 1300 | 1296, H | no |
| 62 | Perilla alcohol | 845, 4.82 | 1300 | 1295 | no |
| 63 | 2-Undecanol | 855, 3.16 | 1306 | H | no |
| 64 | Methyl decanoate | 885, 3.48 | 1325 | 1326, H | no |
| 65 | Methyl geranate | 885, 4.19 | 1325 | 1323 | yes |
| 66 | Ylangene | 945, 3.42 | 1362 | 1372 | no |
| 67 | β -Bourbonene | 965, 3.47 | 1375 | 1385 | no |
| 68 | Vanillin | 985, 7.52 | 1388 | 1402 | yes |
| 69 | 1-Tetradecene | 995, 2.55 | 1394 | 1392, H | yes |
| 70 | Tetradecane | 1005, 2.42 | 1400 | 1400, H | yes |
| 71 | 2-Dodecanol | 1005, 3.16 | 1400 | H | yes |
| 72 | Methyl undecanoate | 1045, 3.32 | 1429 | 1425, H | no |
| 73 | α -Bergamotene | 1055, 3.28 | 1436 | 1436 | no |
| 74 | α -Himachalene | 1055, 3.96 | 1436 | 1447 | yes |
| 75 | β -Ionone | 1115, 4.82 | 1479 | 1482 | no |
| 76 | β -Selinene | 1125, 4.25 | 1486 | 1485 | no |
| 77 | 1-Pentadecene | 1135, 2.83 | 1493 | H | yes |
| 78 | 2-Tridecanone | 1145, 3.50 | 1500 | 1496, H | no |
| 79 | γ -Muurolene | 1145, 4.00 | 1500 | 1477 | no |
| 80 | 2-Tridecanol | 1155, 3.37 | 1507 | H | no |
| 81 | Calamenene | 1175, 4.67 | 1521 | 1521 | no |
| 82 | α -Muurolene | 1185, 4.17 | 1529 | 1499 | no |
| 83 | Caryophyllenyl alcohol | 1225, 4.57 | 1557 | 1568 | no |
| 84 | Caryophyllene oxide | 1245, 4.90 | 1571 | 1581 | no |
| 85 | Hexadecene | 1275, 2.65 | 1593 | 1593, H | yes |
| 86 | 2-Tetradecanone | 1275, 3.59 | 1593 | H | no |
| 87 | Hexadecane | 1285, 2.51 | 1600 | 1600, H | no |
| 88 | 2-Tetradecanol | 1285, 3.38 | 1600 | H | no |
| 89 | Cubenol | 1305, 4.60 | 1615 | 1642 | no |
| 90 | δ -Cadinol | 1335, 4.92 | 1638 | 1658 | no |
| 91 | α -Eudesmol | 1345, 4.77 | 1646 | 1652 | no |
| 92 | α -Cadinol | 1345, 4.98 | 1646 | 1653 | no |
| 93 | Cadalene | 1365, 5.68 | 1662 | 1674 | yes |
| 94 | Juniper camphor | 1395, 5.23 | 1685 | 1691 | no |
| 95 | 1-Heptadecene | 1405, 2.68 | 1692 | H | yes |
| 96 | 2-Pentadecanone | 1405, 3.66 | 1692 | 1700, H | no |
| 97 | Pentadecane, 2,6,10,14-tetramethyl- | 1415, 2.44 | 1700 | 1705 | yes |
| 98 | Heptadecane | 1415, 2.54 | 1700 | 1700, H | yes |
| 99 | Methyl tetradecanoate | 1435, 3.50 | 1717 | 1706, H | yes |
| 100 | <i>cis</i> -Farnesol | 1435, 4.34 | 1717 | 1697 | no |
| 101 | Tetradecanoic acid | 1495, 3.65 | 1767 | 1767, H | no |
| 102 | 1-Octadecene | 1525, 2.73 | 1792 | 1793, H | yes |
| 103 | Octadecane | 1535, 2.60 | 1800 | 1800, H | no |
| 104 | 2-Hexadecanone | 1535, 3.50 | 1800 | 1806, H | no |
| 105 | Methyl pentadecanoate | 1565, 3.43 | 1825 | 1826, H | yes |
| 106 | 2-Pentadecanone, 6,10,14-trimethyl- | 1585, 3.35 | 1842 | 1847 | yes |
| 107 | Pentadecanoic acid | 1605, 3.70 | 1858 | 1839, H | yes |
| 108 | 1-Nonadecene | 1645, 2.76 | 1892 | H | yes |
| 109 | Nonadecane | 1655, 2.63 | | 1900 | no |
| 110 | 2-Heptadecanone | 1655, 3.53 | 1900 | 1910, H | no |
| 111 | Methyl hexadecanoate | 1685, 3.42 | 1927 | 1927, H | yes |
| 112 | Hexadecanoic acid | 1725, 3.73 | 1964 | 1966, H | no |
| 113 | Eicosane | 1755, 2.81 | 1991 | H | yes |
| 114 | Eicosane | 1765, 2.67 | 2000 | 2000, H | yes |
| 115 | Heneicosane | 1875, 2.70 | 2100 | 2100, H | yes |
| 116 | Docosane | 1975, 2.75 | 2200 | 2200, H | yes |

Adapted from: Eyres, G.; Dufour, J. Hop Essential Oil: Analysis, Chemical Composition and Odor Characteristics. In *Beer in Health and Disease Prevention*; Preedy, V. R., Ed.; Academic Press: San Diego, 2009; pp. 239-254.

Experimental

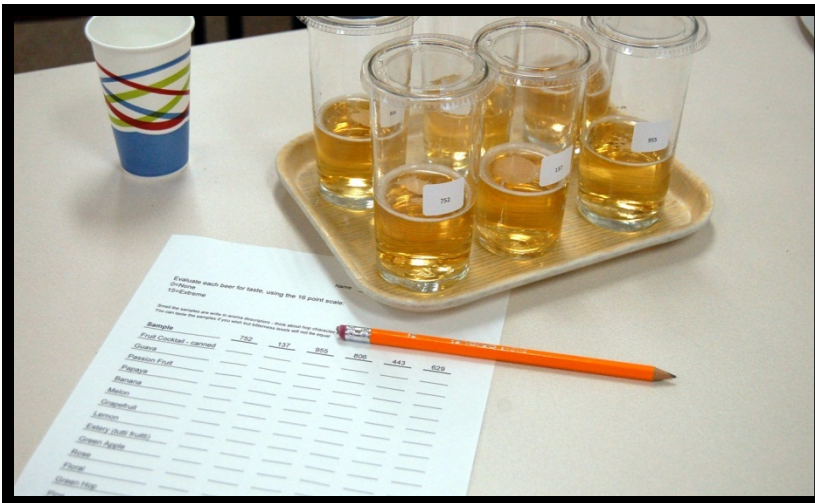
- Hop Acids
 - Composition
 - Hop Storage Index
- Essential Oil
 - Total
 - Composition
- Moisture
 - Post kilning
- **Sensory**
 - **Difference**
 - **Acceptance**
 - **Preliminary Descriptive**



Sensory – 2010 Cascade

Difference Testing: n = 18

- Triangle Test
 - 14 correctly identified different sample ($p < 0.001$)



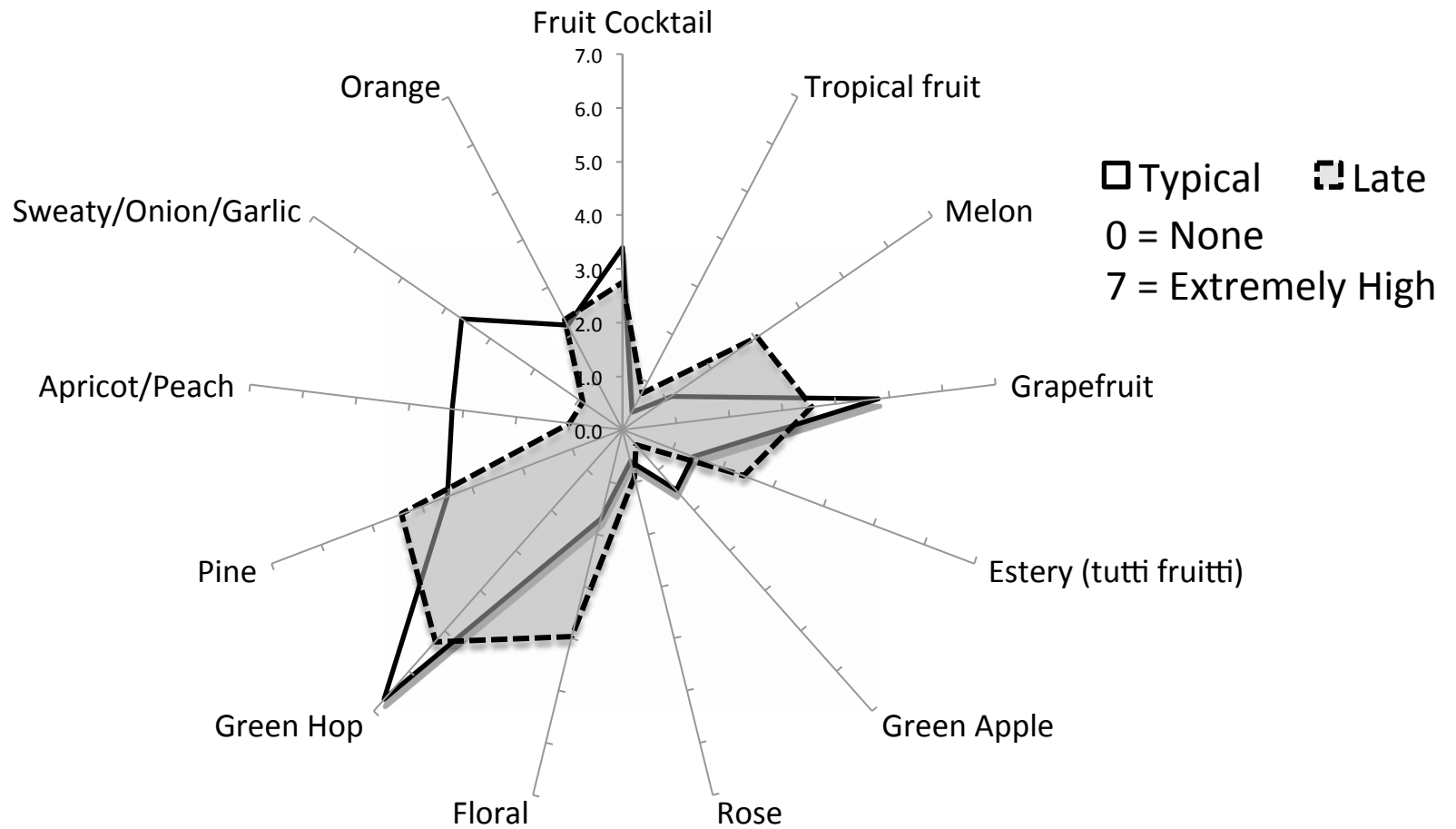
Consumer Testing: n = 62

- Consumer Acceptance
 - Typical preferred over Late (9 point scale)

| <i>Attribute</i> | <i>Typical</i> | <i>Late</i> |
|--------------------|----------------|----------------|
| Overall *** | 7.11 (0.83) | 6.26 (1.61) |
| Aroma *** | 6.92 (1.31) | 5.82 (1.96) |
| Flavor ** | 6.98 (1.03) | 6.23 (1.68) |

***, ** significant at $p < 0.01$ and < 0.05 respectively, (SD),

Descriptive Analysis: 2010 Cascade



Conclusions

- Essential oil content increases past typical commercial harvest dates for Willamette hops, but remain constant for Cascade.
- An increase in oil quantity was strongly correlated with α -pinene, β -pinene, myrcene, limonene, methyl-heptanoate, and linalool.
- Beers brewed with typical harvested Cascade hops were significantly distinguishable in sensory analysis and preferred consumers over late harvested cascade hops.



What next?

- Aroma fingerprinting
 - Degradation indicators?
 - Sulfur compounds
- Predictor variables
- Glycosides



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- OSU Hop Research Farm
- Indie Hops
- Deschutes Brewery

