

## 1. Rahr Corporation, Shakopee, MN; 2. Grist Analytics, Boulder, CO

### Introduction

The increased popularity of hop forward aromatic and flavorful beers has made the method of dry-hopping common practice among the craft brewing industry. Although there are many techniques for dry hopping, potential implications such as increases in diacetyl, over attenuation, increased oxidation rates and other potential negative effects have been associated with the method. There is also the concern of food safety with many products in the craft industry currently being produced above the micro danger zone pH of 4.6.

The objective of the study is to evaluate the analytical impact that pH adjustment may have on a product post dry hopping.

### Method

#### Wort Production

1. A 211 L batch of 12.75 °P wort was produced using a 4 vessel 3 HL brew house. Following cooling of the wort using a heat exchanger, the wort was injected with sterile oxygen to obtain a level of 17 ppm in solution. The higher level of injection was used to compensate for any flash off of O<sub>2</sub> in solution from the wort during the mixing and wort transfer between vessels.
2. All the cooled wort was collected into a single cylindrical conical vessel with racking arm attached to a recirculation loop. The unpitched wort was recirculated for 10 min to ensure a homogeneous sample.
3. Grainfather conical fermenters were then filled to the 6 gallon graduation via weight.
4. The manufacturer recommended dose rate for Fermentis SafAle S-33 of 80 g/hL (18.16 g) was weighed into sterile flasks and pitched directly into each individual fermenter. The yeast was stirred into solution for 10 sec using a sanitized stainless stir rod.
5. Airlocks of PAA were attached to the tops of all fermenters and a glycol unit for temperature control was connected.

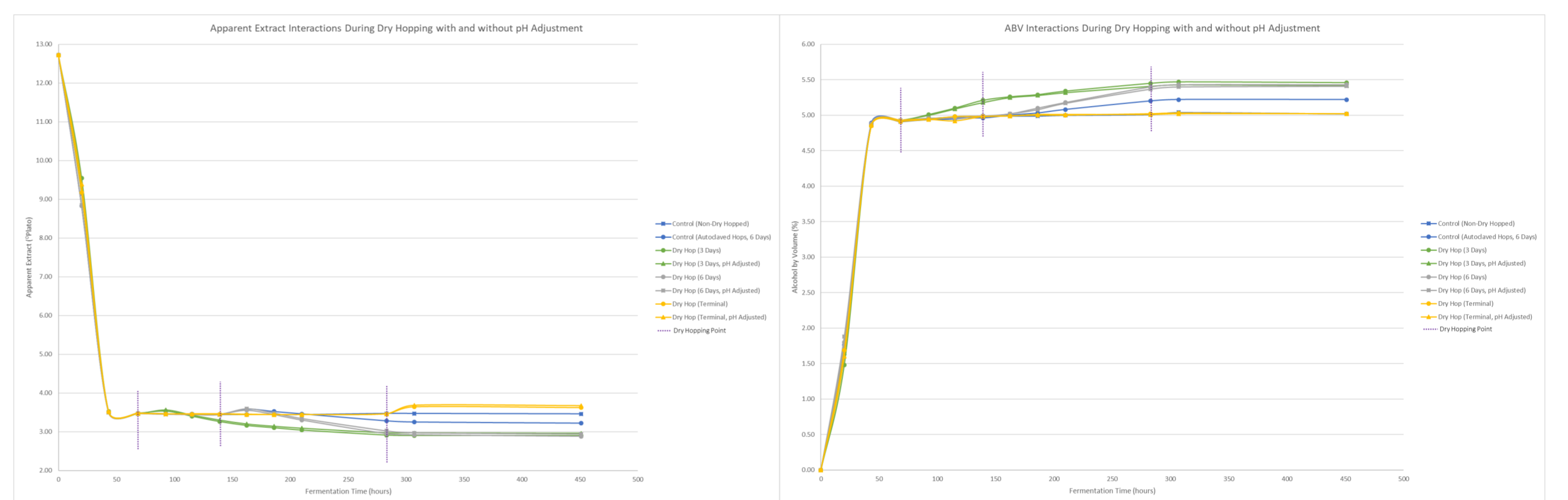
#### Fermentation

1. Fermenters were set to 65 °F. Should be noted that a due technical difficulties a fermenters uniformly increased above the desired fermentation set point during the first day of fermentation which was corrected and brought back into spec.
2. Both dry hopping and acid additions were conducted near the designated residency time of 72 hrs., 144hrs and terminal plato. 75% food grade phosphoric acid was used to perform pH decrease.
3. Samples were dry-hopped at a rate of 2 lbs/bbl and the autoclaved hops were autoclaved at 212 °F
4. Fermentation samples were collected for daily analysis and manual titrations were conducted on samples to determine appropriate acid addition based on sample volume remaining in the fermentation vessels.

\*Volume Remaining was calculated by beginning with the starting weight of wort added to each fermenter and subtracting the volume removed from each fermenter during sample collection, including the volume removed when dropping yeast at Day 6.  
While the starting weight in kilograms doesn't directly equal the volume in liters present in each fermenter, by determining the volume subtracted from the fermenters each day, we could precisely log each fermenter based on the approximate volume remaining (7.5 lbs of hops per barrel). While the accuracy of 2 lbs of hops per barrel wasn't certain, we maintained precision in the concentration of hops added.

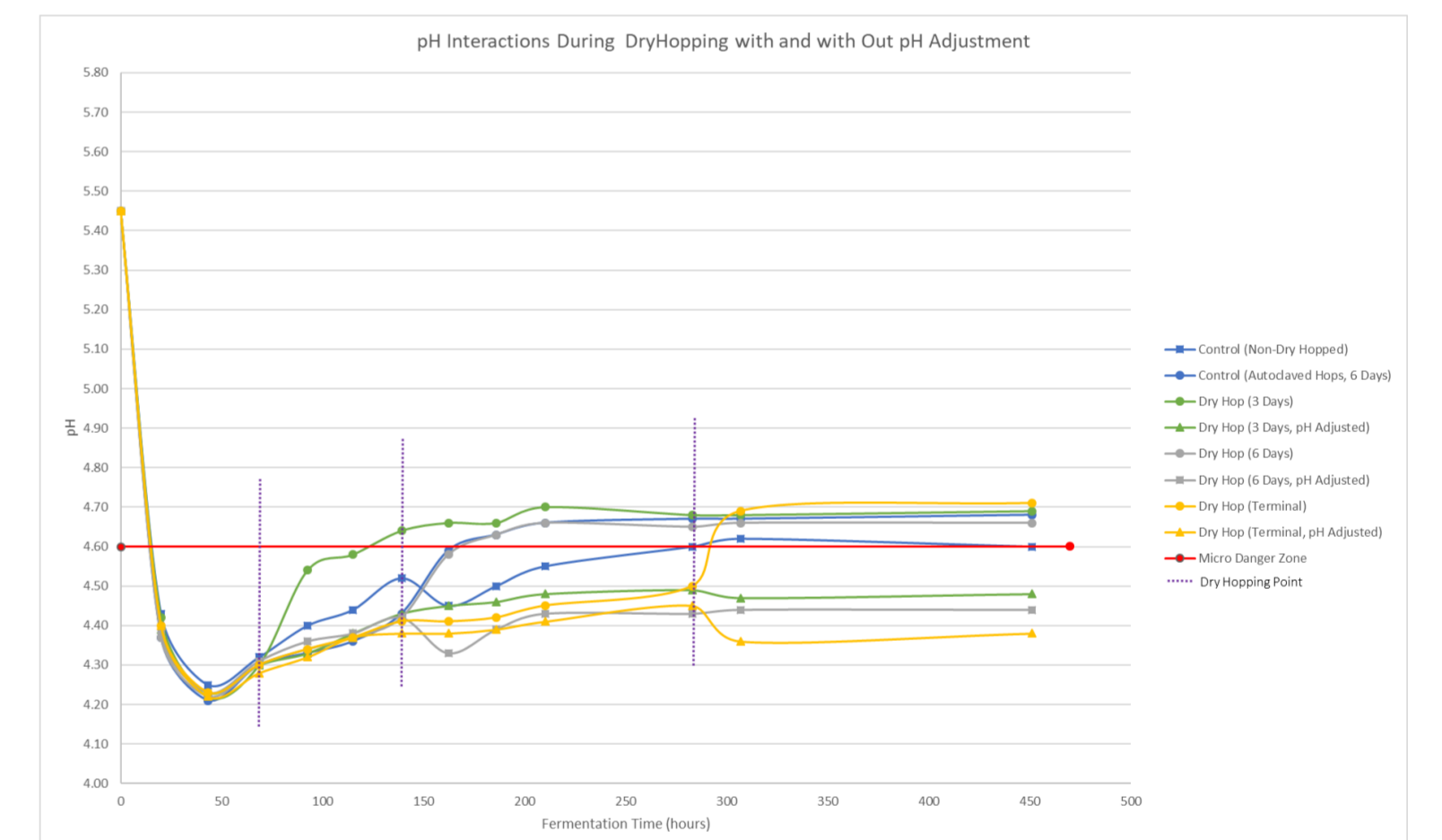
#### Alcohol by Volume (%) and Apparent Extract (° Plato) Interactions

- Increased attenuation and alcohol production occurred during the dry-hopping regime at 3 and 6 days into the fermentation residency.
- No increase was noted in dry-hopping that occurred at terminal although a significant increase in apparent extract (AE) of approx. 0.2 ° Plato was witnessed. A similar increase of approx. 0.1 ° Plato also developed with the day 3 and 6 dry-hopping, although it was followed by increased attenuation.
- This trend was also seen with the 6 day autoclaved dry-hopping control although not to the extent of the regular 3 and 6 day hopping. Both ABV and AE similarly stabilized for pH and non pH adjusted 3 and 6 day samples.
- PH adjustment showed no differentiation among ABV production and attenuation.



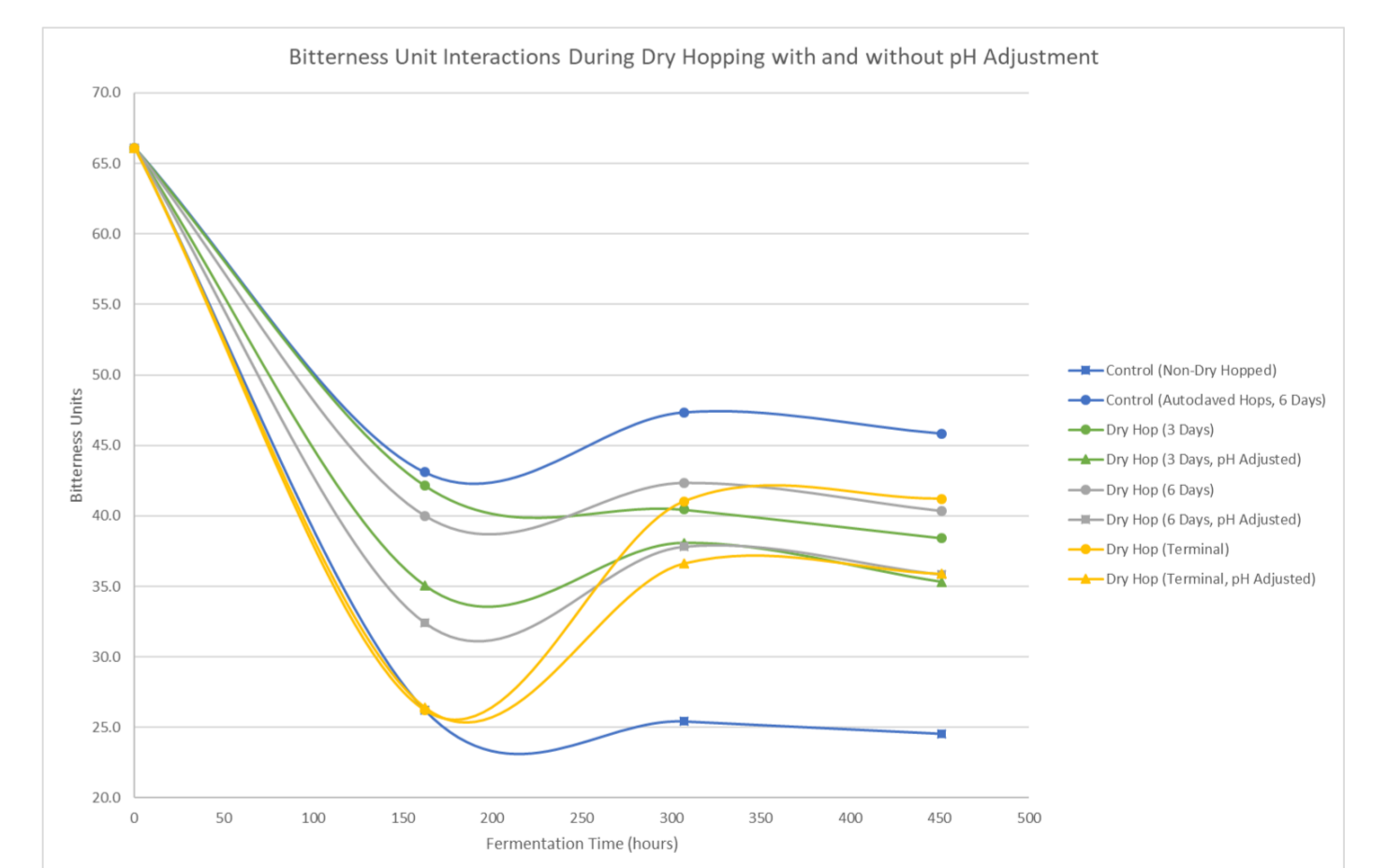
#### pH Interactions

- Dry-hopping can create an increased potential for microbial issues by pushing product into the micro danger zone for pH above 4.6.
- PH adjustment post dry-hopping displayed a greater finished pH impact the later the dry-hopping was performed.



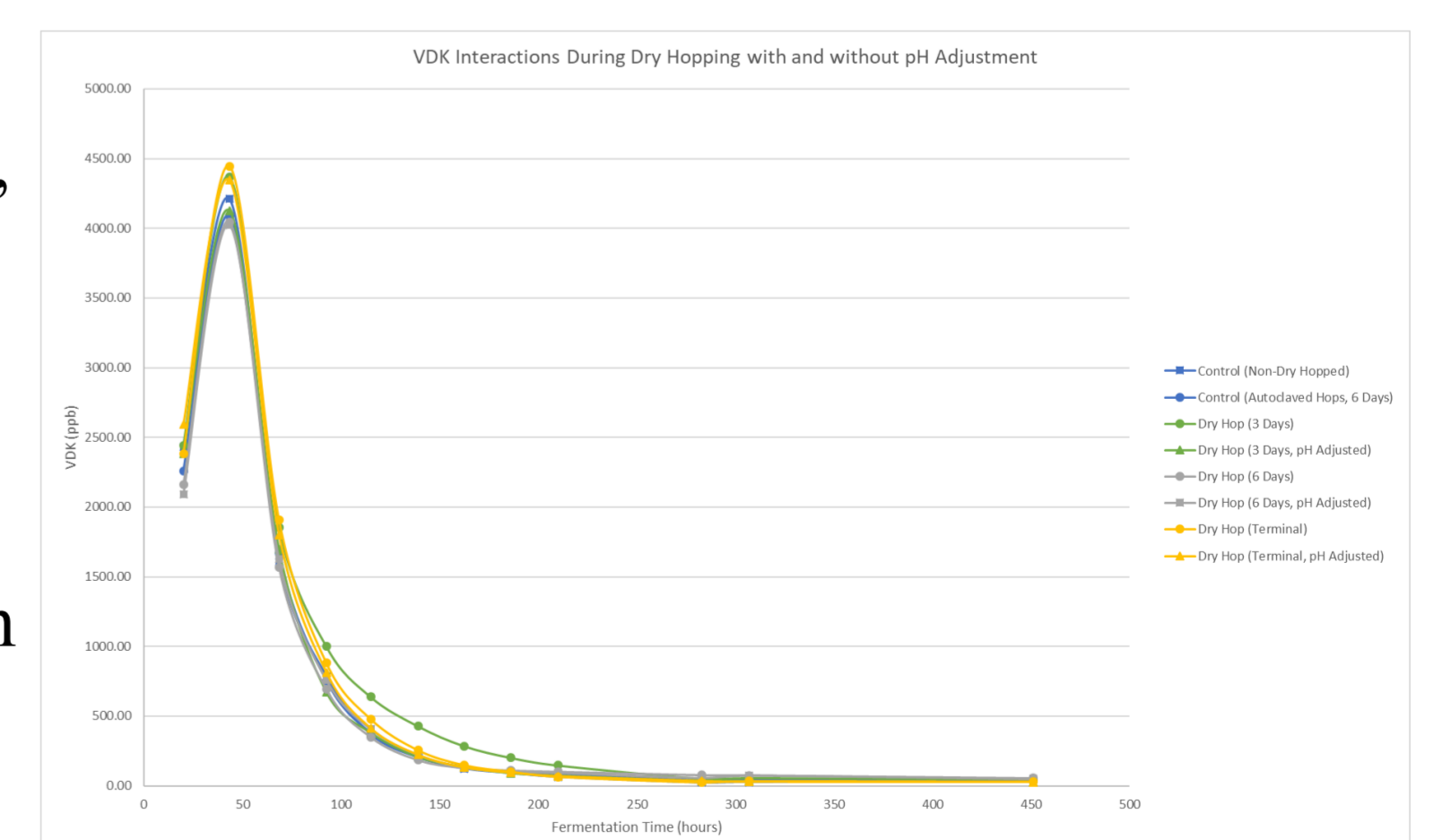
#### Bitterness Unit (BU's) Interactions

- Increased levels of BU's are seen with the autoclaved hop control.
- Dry-hopping resulted in all samples having increased BU's although there was a greater differentiation with samples that had not undergone a pH adjustment. Although also increasing in BU's the three pH adjusted samples finished at a similar BU level.

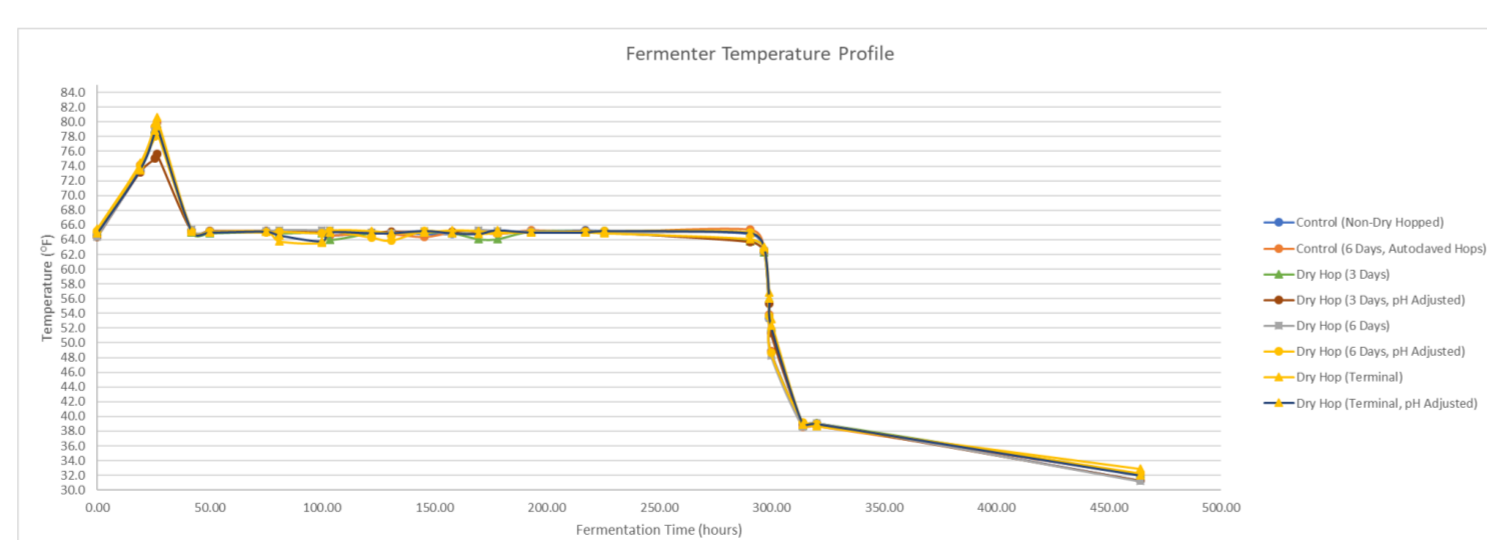


#### VDK Interactions

- Although all tests resulted in a below target threshold of < 80 ppb, it can be noted that the earlier dry-hopping resulted in a slower uptake.
- PH adjusted samples resulted in slightly lower final values than non adjusted samples.



Time in Fermentation (hours)	Fermentation Temperature (°F)									
	Control (Non-Dry Hopped)	Control (6 Days, Autoclaved Hops)	Dry Hop (3 Days)	Dry Hop (3 Days, pH Adjusted)	Dry Hop (6 Days)	Dry Hop (6 Days, pH Adjusted)	Dry Hop (Terminal)	Dry Hop (Terminal, pH Adjusted)	Dry Hop (Terminal, pH Adjusted)	Dry Hop (Terminal, pH Adjusted)
0:00	64.3	64.9	65.2	64.4	64.4	65.2	65.5	64.9		
19:00	73.5	74.2	73.5	73.2	73.6	74.1	74.4	73.5		
25:75	78.5	79.3	78.9	75.0	78.8	78.0	79.8	78.8		
26:50	79.1	79.9	79.5	75.6	79.3	78.6	80.6	79.4		
42:00	65.3	65.2	65.0	65.1	65.4	65.1	65.2	65.1		
50:00	65.1	65.0	64.9	65.2	65.1	65.1	65.1	65.0		
75:00	65.2	65.1	65.1	65.2	65.2	65.0	65.1	65.1		
81:00	65.2	65.1	64.9	65.2	65.3	65.1	65.3	64.6		
99:75	65.0	65.0	64.9	65.0	65.3	64.9	65.6	63.8		
103:50	64.6	64.6	64.0	65.1	65.3	65.2	65.2	65.0		
122:00	64.9	64.9	64.9	65.0	65.0	64.3	65.2	64.9		
130:75	65.0	64.8	65.0	65.1	64.9	65.9	64.9	64.9		
145:50	64.8	64.4	65.0	65.1	65.2	65.2	65.0	65.2		
157:75	64.8	65.0	64.9	65.1	64.8	64.9	65.3	64.9		
169:50	65.2	65.1	64.1	65.1	65.3	64.8	65.2	64.8		
178:00	64.9	64.8	64.1	65.0	65.2	64.9	65.0	65.3		
193:00	65.1	65.3	65.1	65.0	65.1	65.0	65.0	65.0		
217:25	65.3	65.0	65.2	65.2	65.1	65.1	65.0	65.0		
225:50	65.2	65.0	65.0	65.0	64.9	65.2	64.9	65.2		
290:50	64.8	65.4	63.8	63.7	64.9	65.0	64.1	64.9		
296:75	62.3	62.6	62.3	62.4	62.5	62.5	63.0	63.0		
299:00	53.3	53.8	56.0	55.3	53.4	53.6	56.8	56.0		
299:75	48.8	48.8	52.1	51.3	48.3	48.6	53.2	52.3		
314:00	39.1	38.6	38.8	39.0	38.6	39.2	38.9	38.9		
320:00	39.1	38.7	39.1	39.0	39.1	38.7	39.0	39.0		
464:00	31.3	32.1	32.2	31.3	31.2	32.3	32.9	32.0		



Adjusted Grainfather fermenter temperatures from 65°F to 39°F and placed in walk-in cooler at 15:30 on 20-Jun (296.50 hours into fermentation).

Turned off Grainfather glycol chillers at ~320 hours and allowed samples to equilibrate to walk-in cooler temperature

### Results and Data

Key analytics including Alcohol by Volume (%), pH, VDK, Apparent Extract, Bitterness Units and Amylase activity were performed throughout fermentation. Charts and graphs showing data for day 12.79 of residency in the fermenter was the following day post fast cooling the fermenters and day 18.79 reflects the end of a 7day RUH storage.

Sample	Grainfather Fermenter	Starting Weight (kg)	Pitched Yeast (g)	*Volume Remaining (L)	Time in Fermentation (hours)	Dry Hop Weight (g)	Dry Hop Concentration (lbs per barrel)	Volume of 75% Phosphoric Acid Added (mL)	Before Dry Hop (pH)	After Dry Hop (pH)
Control (Non-Dry Hopped)	FV1	23.070	18.21	-	-	-	-	-	-	-
Control (6 Days, Autoclaved Hops)	FV2	23.070	18.16	20.81	149.00	160.9	2.0	-	4.26	4.37
Dry Hop (3 Days)	FV6	23.090	18.16	22.31	71.50	172.5	2.0	-	4.26	4.37
Dry Hop (3 Days, pH Adjusted)	FV8	23.075	18.16	22.31	71.50	172.5	2.0	4.4	4.27	4.14
Dry Hop (6 Days)	FV3	23.045	18.19	21.37	149.00	165.2	2.0	-	4.26	4.32
Dry Hop (6 Days, pH Adjusted)	FV4	23.045	18.16	21.42	149.00	165.6	2.0	4.9	4.31	4.06
Dry Hop (Terminal)	FV5	23.080	18.19	20.92	288.75	161.7	2.0	-	4.42	4.38
Dry Hop (Terminal, pH Adjusted)	FV7	23.095	18.16	20.8125	288.75	160.9	2.0	6.0	4.41	4.07

### Conclusion

Dry-hopping can have implications on the final analytical parameters of a beer. Adjustments in pH can increase the consistency of increases in BU's and provide a an increased level of microbial stability and food safety. Autoclaved hops will still provide a level of hop creep despite the attempt to denature the enzymes. There is suspect that by autoclaving, some hop isomerization had undergone potentially leading to the highest increase in bitterness units. Finished VDK levels were minimally impacted although the samples that were impacted by further attenuation did result in a lower VDK when pH was adjusted.

