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Introduction

Hop creep is the phenomenon of refermentation in beer after dry hopping and is a major source of production and quality issues for brewers. It is facilitated by the action of diastatic enzymes present in hops. These enzymes are transferred to beer during the dry hopping process and proceed to break down unfermentable dextrins. This creates a new influx of fermentable sugars, leading to continued fermentation by yeast. Specific concerns related to refermentation include protracted diacetyl rests, over-attenuation, and excess carbon dioxide production.

Several factors affecting hop diastatic activity were investigated as part of an ongoing effort by the Yakima Chief Hops R&D team to understand hop creep. Using an HPLC-based method for determining sugar concentrations, diastatic activity was quantified to determine the impacts of these different parameters. The factors studied in the present work include variety, seed content, hop material fraction (lupulin vs. bract), and heat treatment.

Methods

Diastatic activity analysis – Hops samples were incubated in a 2% soluble starch solution containing 1 mg/mL bovine serum albumin for 90 minutes with constant shaking. Samples were centrifuged, diluted in acetonitrile, filtered, and analyzed by HPLC. Separation was performed on a Supelco LC-NH2 HILIC column using 70/30 acetonitrile/water mobile phase (isocratic). Each sample had a paired blank with distilled water in place of the starch solution; this allowed the subtraction of endogenous sugars from the result. Diastatic activity was reported as net ppm maltose produced (sample minus blank). Statistical analysis was performed in Excel.

Varietal effect – 700 bale lots from harvest 2020 were analyzed for diastatic activity. Outliers and lots with seed content exceeding YCH's internal quality threshold were not included in averages.

Seed content – Seeds were collected from a pellet line foreign material trap. A standard curve of seed content was created by weighing specific masses of seed into a low-seed base material (1% seed CTZ).

Lupulin vs. bract – A single lot of CTZ was run through the Cryo pelleting process. Samples were taken of the ground raw hop material, lupulin-enriched and bract fractions.

Heat treatment – Samples were subjected to water baths at temperatures ranging from 140 F to 180 F, as well as a room temperature control. The ground hops were incubated for 10 minutes in 45 mL of water at the appropriate temperature. The sample was then removed and allowed to cool. Once the sample reached room temperature, starch solution was added, and the analysis was carried out as described above.

Results and Discussion

Variety was found to affect diastatic activity. Figure 1 shows a statistical summary of diastatic activity data. These were varieties for which 10 or more data points were available. Cascade was found to have the highest average diastatic activity among these varieties at 1573 ppm net maltose. Only Cashmere hops (not shown) were found to have a higher average diastatic activity at 2096 ppm net maltose, but this was based on a small sample size of 5 bale lots. Other major varieties include Mosaic at 1029 ppm net maltose and Simcoe at 1247 ppm net maltose. Citra stands out as having the lowest average of any major variety studied, at only 457 ppm net maltose.

This data was obtained from a single harvest and should not be viewed as conclusive. Furthermore, results from this analysis may not perfectly reflect each variety's hop creep potential in an actual dry hopping.

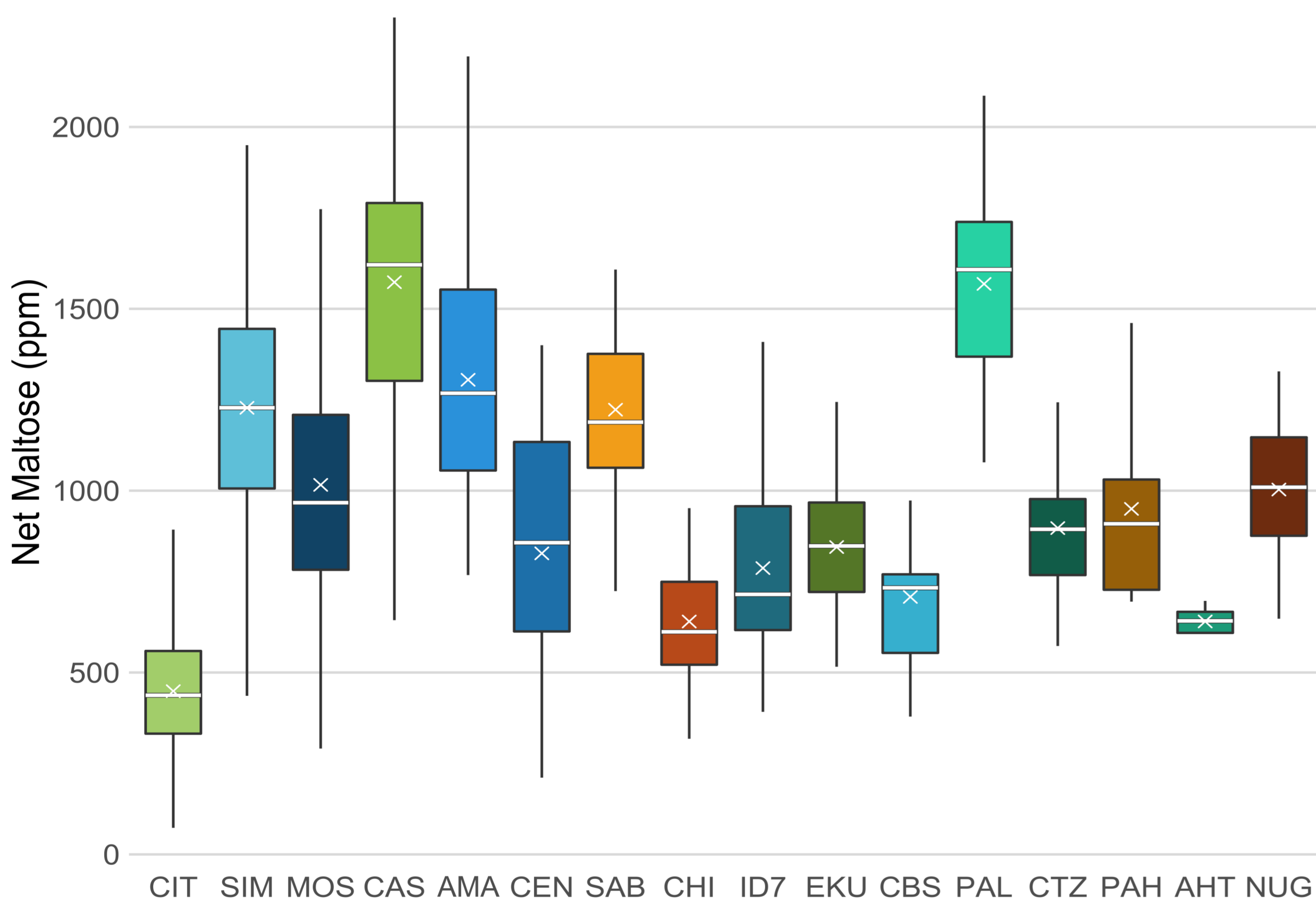


Figure 1. Box and whisker plot for 16 varieties with $n \geq 10$ data points. High-seed samples and outliers were not included. X mark indicates mean value.

Diastatic activity was found to increase with seed content. In the seed addition trials, net maltose production increased linearly with added seed mass up to 10% w/w added seed. An all-seed sample was analyzed and found to produce in excess of 10,000 ppm maltose; this measurement exceeded the calibration range of the instrument and is not shown. An all-seed blank was also analyzed and found to contain less than 100 ppm maltose, indicating that endogenous sugars in the seed are not responsible for the elevated maltose content.

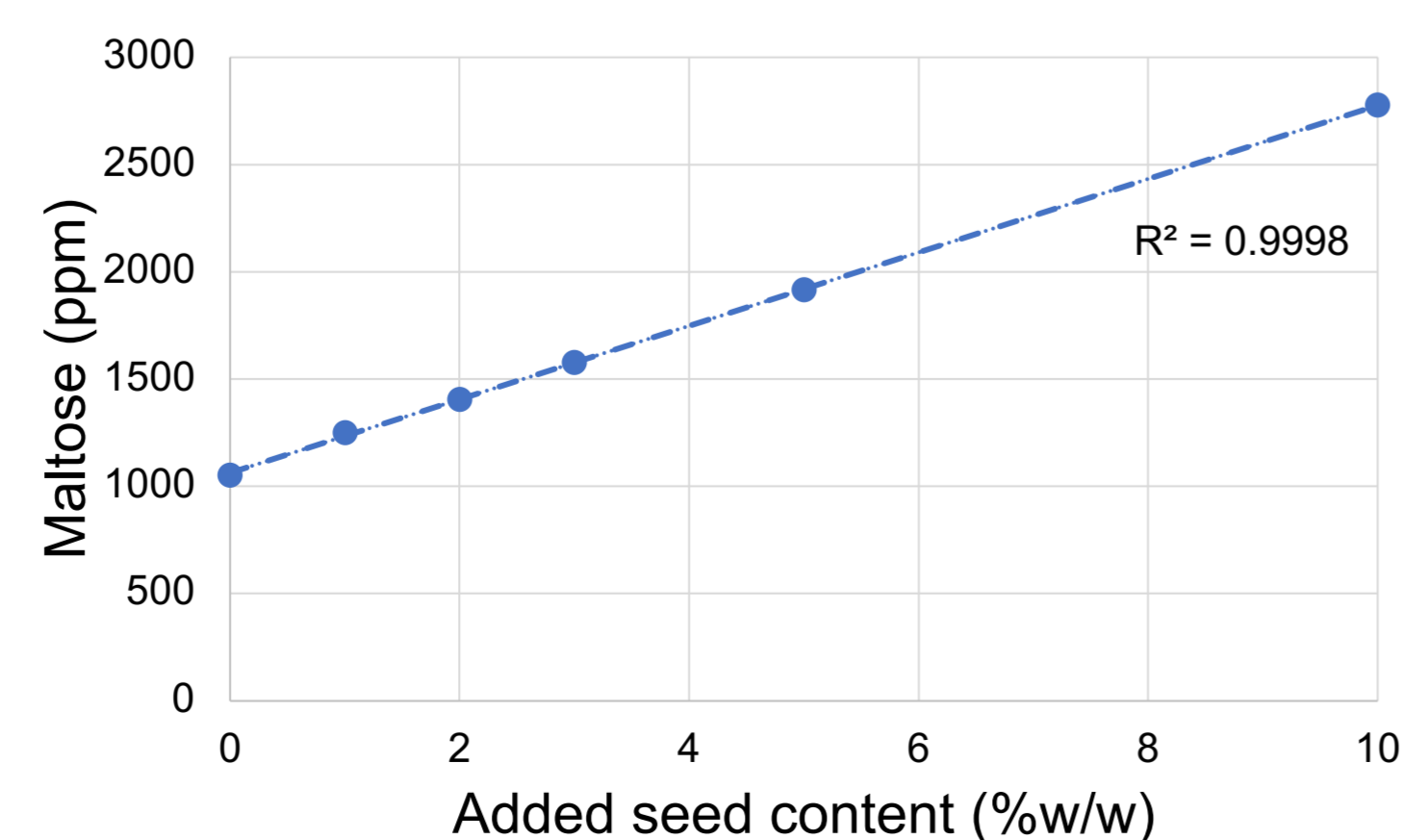


Figure 2. Diastatic activity vs added seed content. All-seed blank (not shown) contained <100 ppm maltose.

Lupulin-enriched material (Cryo) was found to have lower diastatic activity than whole hop or bract fractions. Figure 4 shows net maltose production by each fraction obtained from a Cryo run of CTZ. The Cryo process removes bract, resulting in a lupulin-enriched pellet. The lupulin-enriched fraction exhibited approximately 25% of the diastatic activity of the ground whole hops. Bract material showed elevated diastatic activity – 150% that of the ground whole hops fraction.

The dramatic differences between fractions appear to corroborate the industry assumption that bract contains a greater concentration of diastatic enzymes than lupulin. High seed content in the starting material also likely played a role in these results.

Heat-treating hops reduced their diastatic activity. Incubation at 140 °F for ten minutes resulted in a roughly 50% reduction in diastatic activity compared to a room temperature incubation. Successive temperature increments resulted in lower levels of maltose. Enzymatic activity was negligible after 180 °F treatment.

The Cascade sample retained its diastatic activity better than the other varieties. Maltose production by cascade was reduced by only 38% at 140 °F, compared to >50% for the others. This result may indicate that Cascade's diastatic potential is more robust than other varieties. Meanwhile, Citra's diastatic potential is so low that its endogenous sugars make up a considerable portion of its maltose content in this analysis.

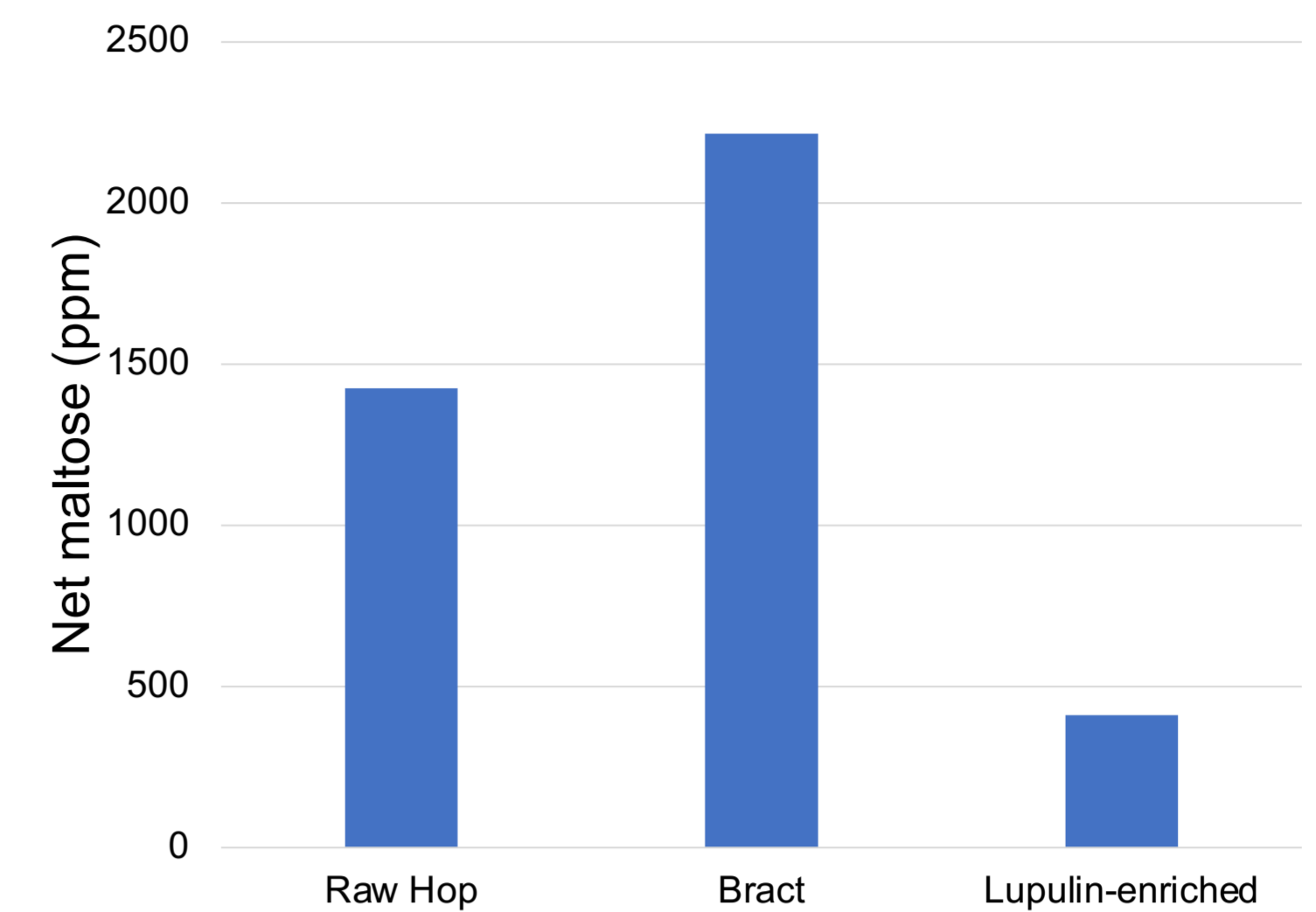


Figure 3. Diastatic activity comparison of raw hop, bract and lupulin-enriched fractions.

Table 1: Effect of Heat Treatment on Diastatic Activity

Temperature (°F)	Diastatic Activity % Change			
	CAS	MOS	SIM	CIT
70	0	0	0	0
140	-38	-51	-53	-53
150	-54	-65	-73	-67
160	-70	-87	-87	-69
170	-87	-94	-91	-76
180	-95	-94	-92	-77

Analysis of the higher-temperature blanks was complicated by the presence of coeluting compounds. Accordingly, total rather than net maltose is shown.

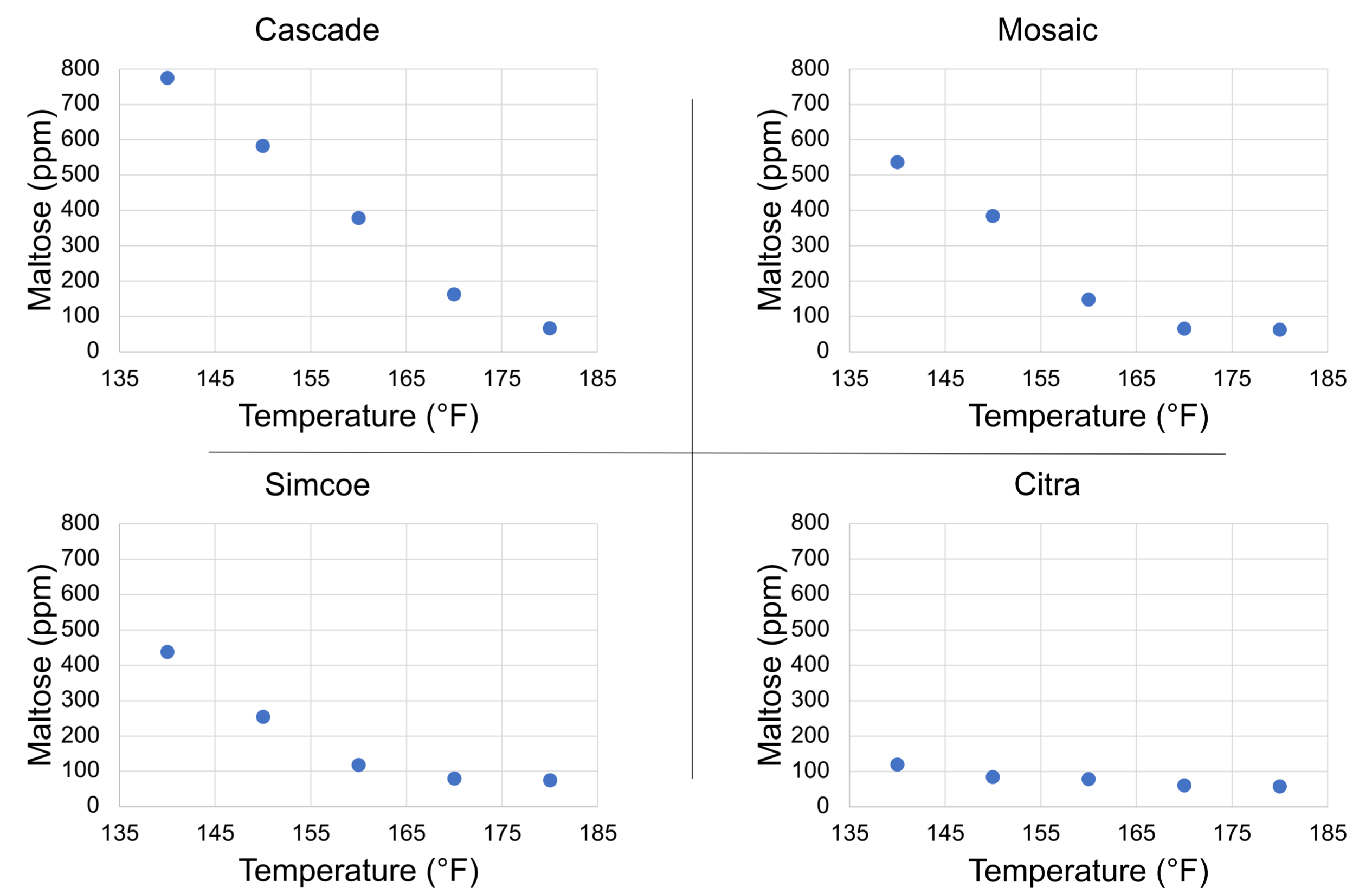


Figure 4. Plots of diastatic activity vs. water bath temperature for four varieties subjected to heat treatment.

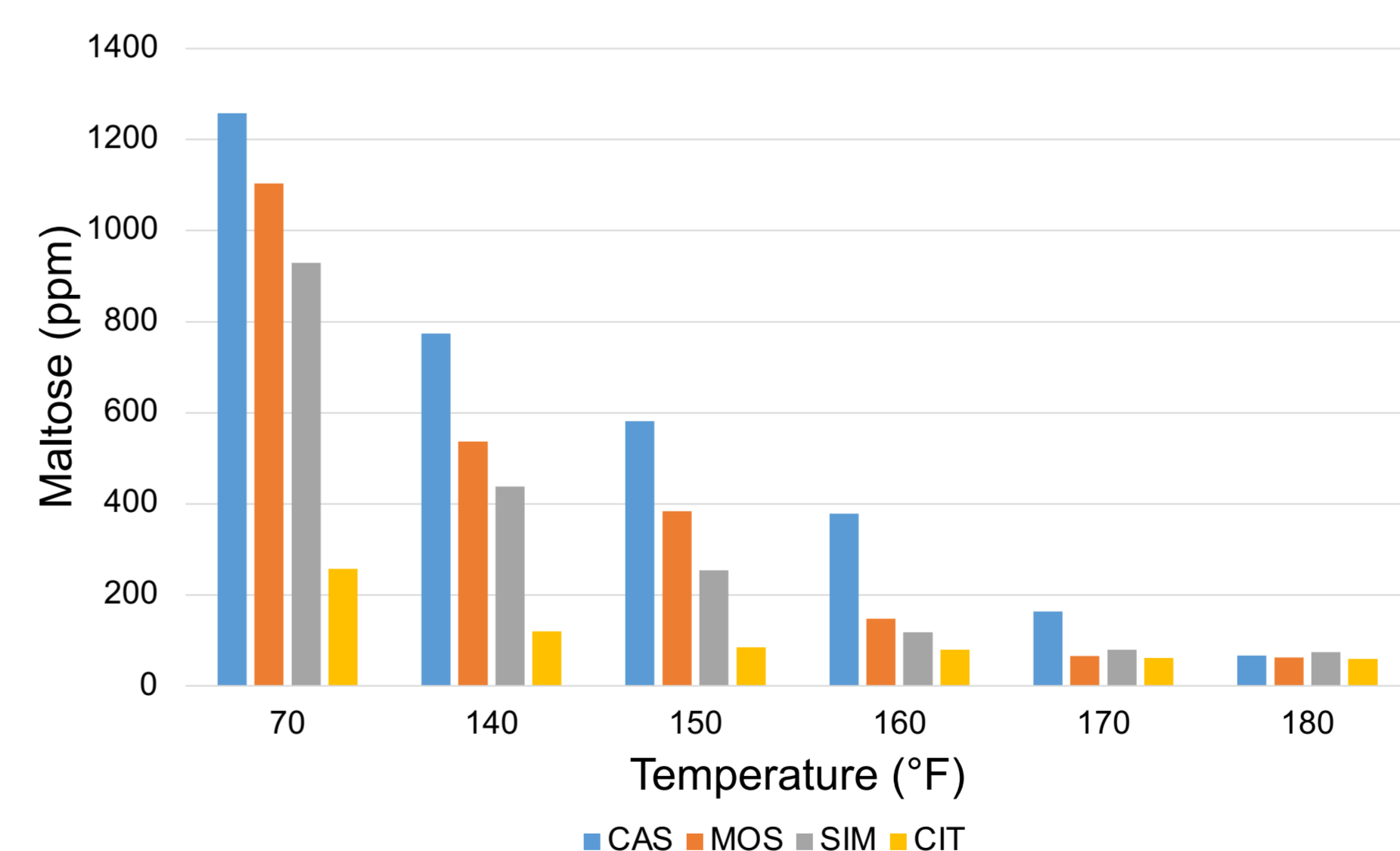


Figure 5. Comparison of diastatic activity by variety per temperature increment.

Conclusion and Future Work

The ability to predict and manage hop creep is a major goal of the brewing industry. This research uses a laboratory method to gather data about diastatic activity in hops. Going forward, diastatic activity data can be used to help predict hop creep severity (broadly by variety or specifically by lot), and to motivate quality practices and production processes (seed content and pellet type). This research also presents proof of concept for a potential brewer practice to diminish diastatic activity, namely heat treatment.

More large-scale studies of diastatic activity in hops are needed to fully determine the role of variety as well as harvest year, climate, and various agronomic practices. More pilot-scale experiments are also needed to demonstrate the efficacy of hop creep mitigation practices such as replacement of T90 with other products, or novel practices such as heat treatment.

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