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Determination of iron concentrations in beer through key steps in the brewing and packaging processes using iron analysis by ferrozine

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OVERVIEW

In an effort to help improve shelf stability and the understanding of how detrimental iron is to the stability of beer, iron concentrations were analyzed at several key points in the production process. Iron in beer can be detrimental to its organoleptic characteristics, stability, and quality. Thus the iron content of beer should be as low as possible. Metallic notes are derived from contamination of beer with metal ions, and metallic odors can also be produced by lipid oxidation. This study focuses on where iron is lost (fermentation, spent grain, trub, yeast) and potentially gained (raw materials, filtration medium, packaging materials) throughout the brewing and packaging processes. Only 2 of the 13 beer styles analyzed were above the industry benchmark value (0.040 mg/L iron) in bottled beer.

MATERIALS & METHOD

ASBC Beer-18C method was used to determine the iron concentration in beer

Calibration: Three standards of known iron concentrations were made following **Table 1**. A plot of absorbance (y-axis) versus added iron (x-axis) was made, reference **Figure 1**, and the slope was calculated by linear regression.
Average Slope = 0.39

Table 1: Preparation of Flasks for Calibration

Standard Flask	Concentration (mg/L)	5 mg/L Standard (Reagent C) (mL)	Water (mL)
1	0.00	0.0	4.0
2	0.10	1.0	2.0
3	0.20	2.0	1.5

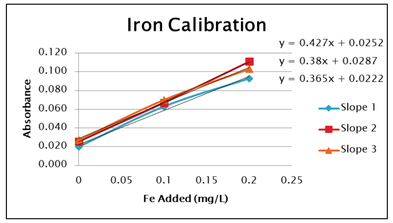

Figure 1: Iron Calibration Curve

Method: Ferric iron (Fe^{3+}) in beer is reduced to the ferrous form (Fe^{2+}) by ascorbic acid. Ferrozine then reacts with the ferrous iron to form a colored complex. The color development is proportional to the ferrous iron present in the sample. Iron concentrations are determined by comparing absorbance at 562 nm with a series of standards.

Calculations: For each sample, subtract the blank absorbance from the corresponding test absorbance. Divide the net absorbance of the sample by the standard calibration slope to give the iron concentration in mg/L.

$$Fe\left(\frac{mg}{L}\right) = \frac{A_t - Ab}{S}$$

A_t= Absorbance of the test
A_b= Absorbance of the blank
S= slope from calibration curve

INTRODUCTION / TYPES OF BEER ANALYZED

- Iron/ BSI (brewing soluble iron) is known to come from raw materials (higher is specialty malts) and possibly filtration medium
- In excess iron can cause metallic flavor (≥0.5ppm)
- In smaller amounts, iron is known to negatively affect shelf stability
  - Iron ions can donate electrons to form free radicals which participate in redox reactions and accelerate loss of fresh beer flavor
- Samples collection points
  - Wort- during knockout (post heat exchange)
  - Post chill- fermentation complete and pre filtered 32-45 ºF beer
  - Bright tank- post centrifuge and filtration
  - Bottle- finished packaged beer

Table 2: Beer Styles Analyzed

Beer Style	Brand
India Pale Ale (IPA)	IPA1, IPA2
Double IPA	DIPA1,DIPA2, DIPA3
Red IPA	RIPA
Black IPA	BIPA
Session IPA	SIPA
American Strong Ale	ASA
American Pale Ale	PALE
Belgian Style IPA	BEL
Multigrain Amber Ale	MAA
Imperial Stout	ISA

RESULTS AND DISCUSSION

Question 1: How do iron concentrations in wort compare to concentrations in finished beer?

- For all beer styles, wort contains a higher concentration of iron compared to the finished beer product, **Figure 2**. All of the raw unfermented products in the wort contribute to this larger amount of iron. Beer styles that contain more specialty malts will also have larger iron concentrations compared to those with non specialty
- A large reduction in iron concentration is shown once the beer has been chilled. Once the beer has been chilled and the yeast has been dumped, this trub waste will contain a larger portion of the iron
- More specifically, the west coast style IPA (1) had iron concentrations of 0.082 mg/L in the wort and 0.040 mg/L of iron in the chilled pre-filtered beer sample

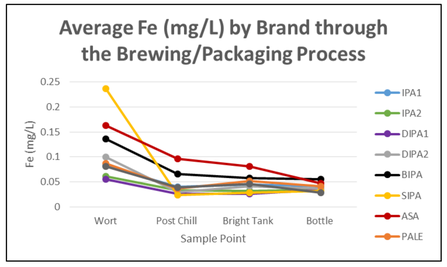

Figure 2: Average Fe (mg/L) by beer style throughout the brewing and packaging process

Question 2: Does the filtration medium, Perlite, add iron to the finished product?

- Previous research suggest that the filtration medium diatomaceous earth (DE), increased the concentration of iron in the finished beer product. To determine if the our filtration medium, Perlite, also increases the iron content we compared beer in the fermenter (post-chill) to beer after it was filtered into the bright tank
- We can conclude that there is no iron pickup from this filtration medium because iron concentrations are the same (or less) pre and post filtration (post chill vs bright tank)

RESULTS AND DISCUSSION

Question 3: Is there any iron pickup from packaging equipment or materials (lines, bottles, crowns)?

- **Figure 2** shows on average there is no iron pickup between the beer in the bright tank and the beer once it has been packaged
- Iron concentrations were compared in rinsed, undecorated bottles to bottles that were prepped and decorated, ready to be filled. There was no difference in the concentrations of these two bottles proving the rinse water nor the decoration ink lead to an increase in iron

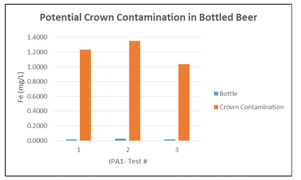

Figure 3: Potential crown contamination in finished beer

- The efficiency of the crown liner was under investigation because a in house study showed (**Figure 3**) that the crown itself could significantly increase the iron in the beer causing shelf stability issues if the crown was in direct contact with the beer
- Figure 3 represents a positive control for this test
- A "crown test" was conducted to determine if the crown liner was sufficient at decreasing the exposure of the beer to the crown. Control bottles were stored upright for 3 days and test bottles were inverted and stored for 3 days
- On average, there was little difference in iron concentration for the control beer vs crown test beer (**Figure 4**)
- T-test confirmed there was no significant difference between the two data sets

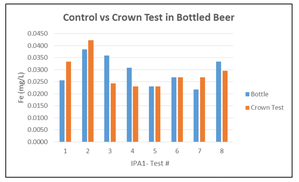

Figure 4: Control vs crown test in bottled beer

Question 4: Do all 13 styles of beer analyzed fall below the industry benchmark values?

- Majority of the beer styles analyzed do fall below the industry benchmark value of 0.040 mg/L iron (**Figure 5**)
- Beer style 7 (black IPA) and 13 (imperial stout) have iron concentrations well above this industry standard. These beers have two things in common: they are darker in color and they use a larger concentration on specialty malts compared to the rest of the beer styles listed
- This indicates that specialty malts may container higher concentrations of iron compared to non specialty malts such as 2-row base malt. This is a future study.

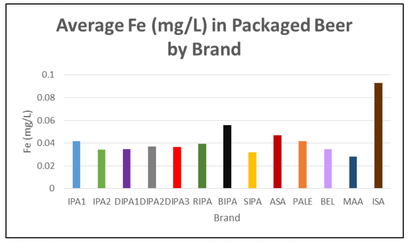

Figure 5: Average Fe (mg/L) in finished beer by style

RESULTS AND DISCUSSION

Question 5: Is there a difference in iron concentrations in fresh beer vs beer stored for 90 days?

- In an effort to help determine a possible cause for shelf stability issues, iron concentrations were compared in fresh beer vs beer stored for 90 days
- According to the t-test there is no significant difference in these two

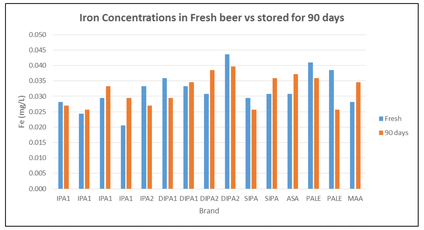

Figure 6: Iron concentrations in fresh beer versus beer stored for 90 days

CONCLUSION

Question 1: How do iron concentrations in wort compare to concentrations in finished beer?

- Wort samples contained a higher concentration of iron compared to finished beer for all styles evaluated in this test. Iron concentrations decrease during the brewing process and are lost to spent grains, trub and yeast

Question 2: Does the filtration medium, Harborlite, add iron to the finished product?

- There was no significant difference in average iron concentrations between pre and post filtered beer which indicates the filter medium, Harborlite, does not add additional iron

Question 3: Is there any iron pickup from packaging equipment or materials (lines, bottles, crowns)?

- There is no iron pickup in the beer from the crowns, therefore, the lining on the inside of the crown is adequate at preventing iron from leaching into the beer. Packaging process nor equipment contributes to an increase in iron for all beer types analyzed

Question 4: Do all 13 styles of beer analyzed fall below the industry benchmark values?

- Majority of the beer styles analyzed do fall below the industry benchmark value of 0.040 mg/L iron. Beer style 7 (black IPA) and 13 (imperial stout) have iron concentrations well above this industry standard

Question 5: Is there a difference in iron concentrations in fresh beer vs beer stored for 90 days?

- According to the t-test there is no significant difference in fresh beer versus beer stored for 90 days at cold temperature

REFERENCES

American Society of Brewing Chemists. *Methods of Analysis*, revised 2015. Beer 18C Iron Analysis by Ferrozine. ASBC, St. Paul, MN.

Bamforth, C. Scientific Principles of Malting and Brewing. Vol. 3, pp. 14, 81, 133, 155, 2012.

McCarthy, S., et al. Iron in Beer by Ferrozine Method. *J. Am. Soc. Brew. Chem.* 49:0177, 1991

Wietstock, P., Kunz, T., Waterkamp, H., and Methner, F. Uptake and Release of Ca, Cu, Fe, Mg and Zn during Beer Production. *J. Am. Soc. Brew. Chem.* 73(2):179-184, 2015.

Zufall, C., and Tyrell, T. The Influence of Heavy Metal Ions on Beer Flavour Stability. *Journal of the Institute of Brewing*. 114(2):134-141, 2008.