Haze Stability and Particle Size Distribution Investigations in NEIPAs

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Abstract

Particle size and distribution measurements were performed using specialized instrumentation (Mastersizer 3000, Malvern Instruments) on a collection of New England Hazy IPAs obtained from the market. This investigation examined the particle size distribution in relation to the haze stability of the commercial products over time. Key properties of the haze particles such as the average particle size or median diameter (D50), volume mean diameter (D [4, 3]), and surface mean diameter (D [3,2]) were measured. The particle size distribution in diameters (µm) for up to 10%, 50% and 90% of the total particles is expressed in D10, D50 and D90, respectively, and was calculated for the beer samples. Variables of pH and effects of filtration and centrifugation impacting the particle size distribution and haze over time were examined. The results and findings provide data and insight into haze stability of NEIPAs through particle size distribution as related to environmental factors and processes. Here we observed the impact of particle size differences and distribution between the most stable and the least stable commercial beer samples.

Experimental Methods

Commercial beer was purchased (6 brands, 12 cans each) for investigations into haze stability in relation to particle size. Beer was cold stored at ~4°C in an upright position and the beers were not disturbed while being opened and poured to keep the sediment and settling representative of the timepoint. Photos were taken at each time point. Two cans of each brand were sent to North Dakota State University (NDSU) for particle size testing on the Mastersizer 3000 with Hydro dispersion system (Malvern Instruments Ltd). The particle size testing was performed at Month 0 and Month 3. Two cans of each brand were tested at Month 0 for haze, pH, pH altered +/- ~0.2 then tested for haze, centrifugation then haze test, and filtration then haze test. All haze measurements were taken via turbidity on the 2100 N IS Turbidimeter. One can was tested at Month 0.5, 1.5, and 3 for haze and pH.

Results & Discussion

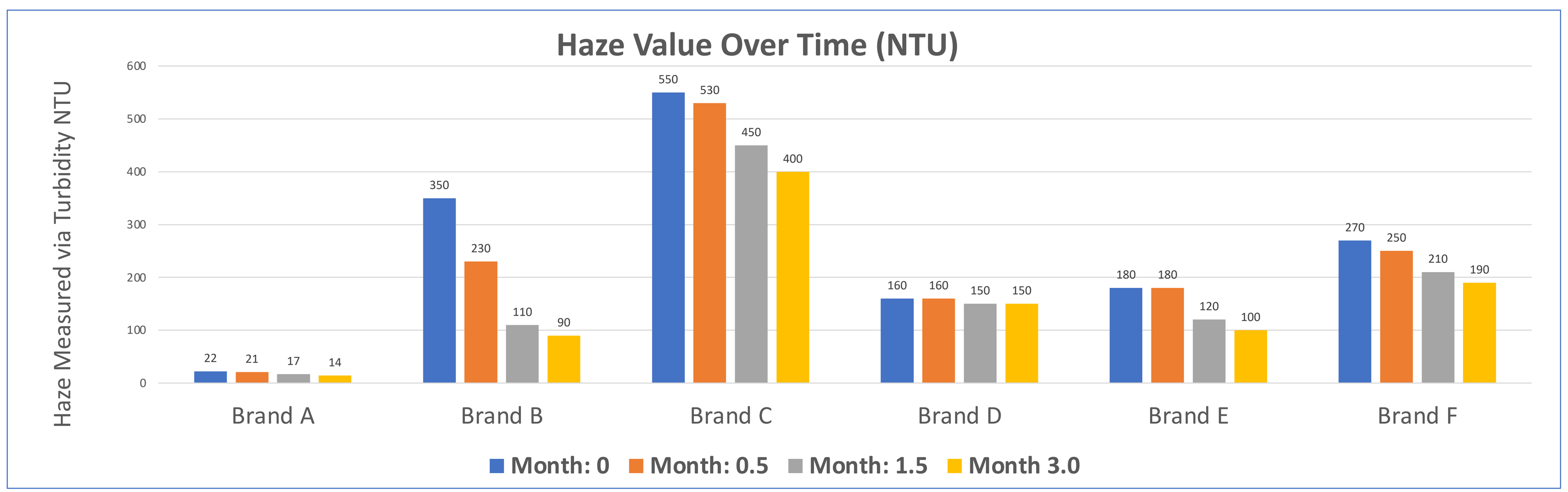

Figure 1: Haze values in NTU were evaluated on six commercial beer samples at 0, 0.5, 1.5, and 3 months.

NDSU PARTICLE SIZE DATA 2/3/2022 - Month 0							
Sample Code	Sample Name	D [3,2]	D [4,3]	Dx (10)	Dx (50)	Dx (90)	Span
1	Brand A	0.07±0.01	42.11±6.28	0.03±0	0.12±0.03	157.88±21.1	1374.65±378.9
2	**Brand B**	**0.08±0**	**13.89±5.62**	**0.03±0**	**0.16±0.01**	**53.88±52.64**	**326.47±315.41**
3	Brand C	0.07±0	4.23±2.97	0.03±0	0.13±0	0.85±0.2	6.46±1.45
4	**Brand D**	**0.22±0.02**	**43.53±5.17**	**0.09±0.01**	**0.64±0.07**	**169.5±7.97**	**268.44±28.1**
5	Brand E	0.08±0	6.88±3.67	0.03±0	0.16±0.01	2.27±1.03	13.74±5.86
6	Brand F	0.07±0	7.66±1.47	0.03±0	0.12±0	3.37±0.78	27.52±5.49

Table 1: Month 0 particle size distribution data. The particle sizes are in micrometers. D [3,2] is called the Sauter Mean Distribution or the surface area distribution mean value. It is sensitive to smaller particles as there is more surface area than volume in a small particle. D [4,3] is called the De Brouckere mean or the volume mean diameter. This measurement is sensitive to larger particles as there is more volume in a large particle compared to the surface area. Dx (10), Dx (50), and Dx (90) indicate the size of particles that land within 10%, 50%, and 90% of the sample. The span gives indication how far apart the 10% and 90% points are, normalized with a midpoint.

NDSU PARTICLE SIZE DATA 4/29/2022 - Month 3.0							
Sample Code	Sample Name	D [3,2]	D [4,3]	Dx (10)	Dx (50)	Dx (90)	Span
1	Brand A	0.07±0.01	51.38±20.21	0.03±0	0.12±0.03	205.17±53.62	1643.80±221.22
2	**Brand B**	**0.06±0**	**30.95±8.43**	**0.02±0**	**0.10±0.01**	**161.83±33.61**	**167.64±314.36**
3	Brand C	0.06±0	8.45±6.59	0.02±0	0.09±0	0.35±0.02	3.70±0.16
4	**Brand D**	**0.17±0.02**	**21.96±2.33**	**0.06±0.01**	**0.43±0.02**	**113.20±13.58**	**267.86±44.58**
5	Brand E	0.06±0	44.72±16.38	0.02±0	0.09±0	0.57±0.15	5.96±1.56
6	Brand F	0.07±0	25.72±8.15	0.03±0	0.11±0.01	121.28±31.63	1079.43±226.84

Table 2: Month 3 particle size distribution data.

The highlighted data in Tables 1 and 2 for Brands B and D produced an interesting result. Brand B haze values went down 75% over the course of three months. Large particles dropped out over time due to coagulation of polyphenols and proteins and the process of sedimentation. D [4,3] has a larger value in Month 3, but knowing most of the haze dropped out, it shows the remaining particles to be coagulating and forming larger particles over time, which then eventually drop out. The Dx (50) value for Brand B drops, indicating 50% of the particles fall into this smaller size at Month 3. The change in haze is also obvious in the visual photos taken at Month 0 and 3, see Figures 2 And 3. Brand D was remarkably stable, having only lost a little more than 6% over three months. The D [4,3] value for Brand D does decrease, indicating the largest particles did fall out and the small particles are quite stable since the haze values did not drop much over three months.

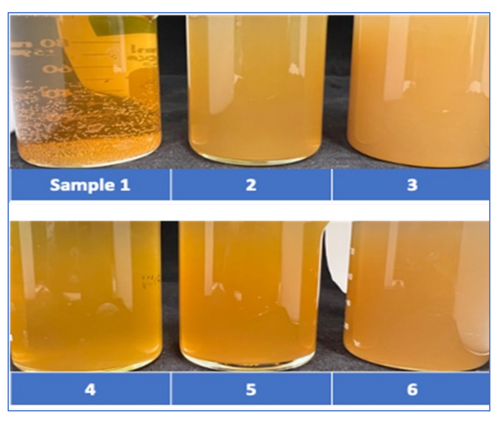

Figure 2: Month 0

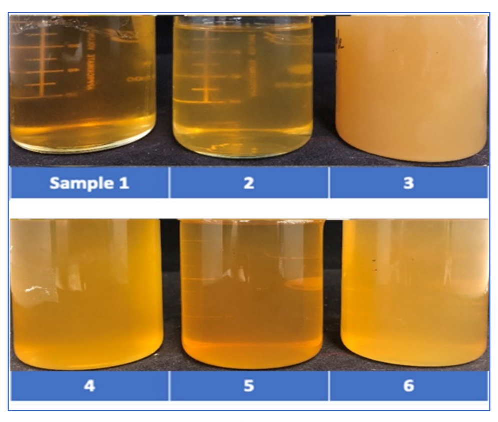

Figure 3: Month 3

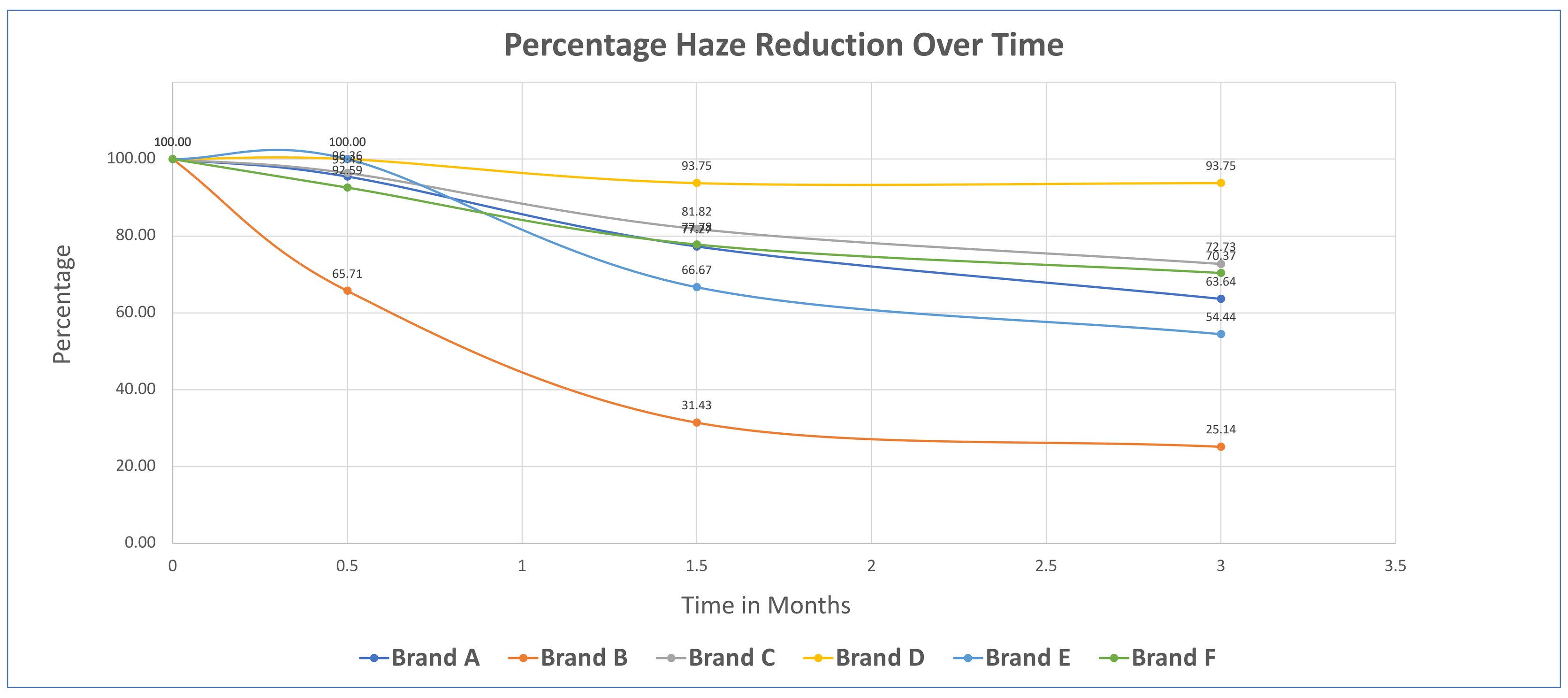

Figure 4: Haze NTU values over three months calculated into percentage reduction loss.

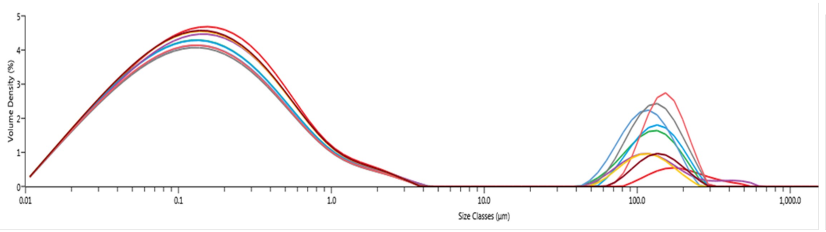

Figure 5: Month 0 Brand B Particle Size Distribution

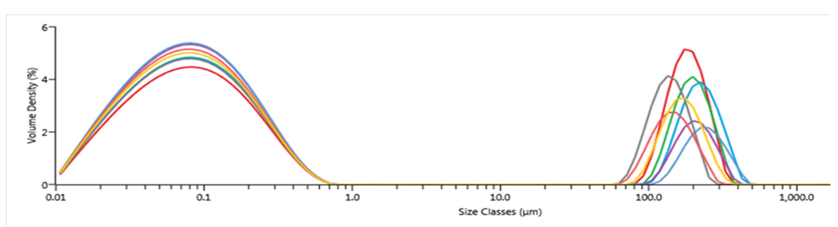

Figure 6: Month 3 Brand B Particle Size Distribution

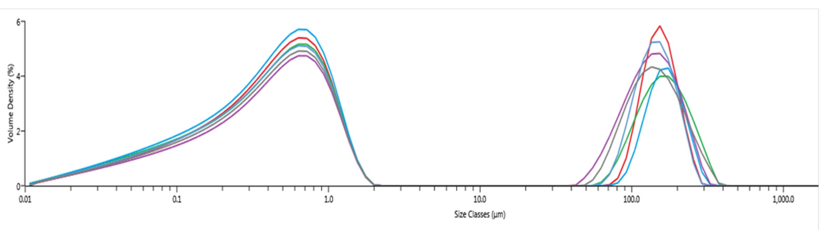

Figure 7: Month 0 Brand D Particle Size Distribution

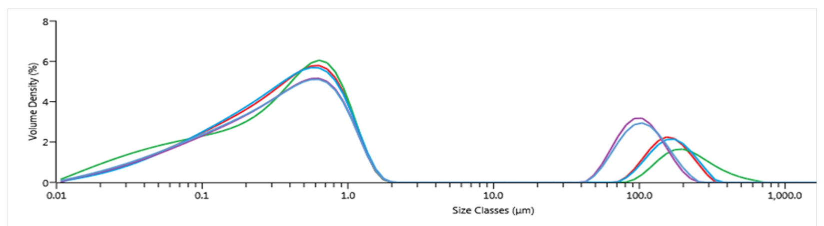

Figure 8: Month 3 Brand D Particle Size Distribution

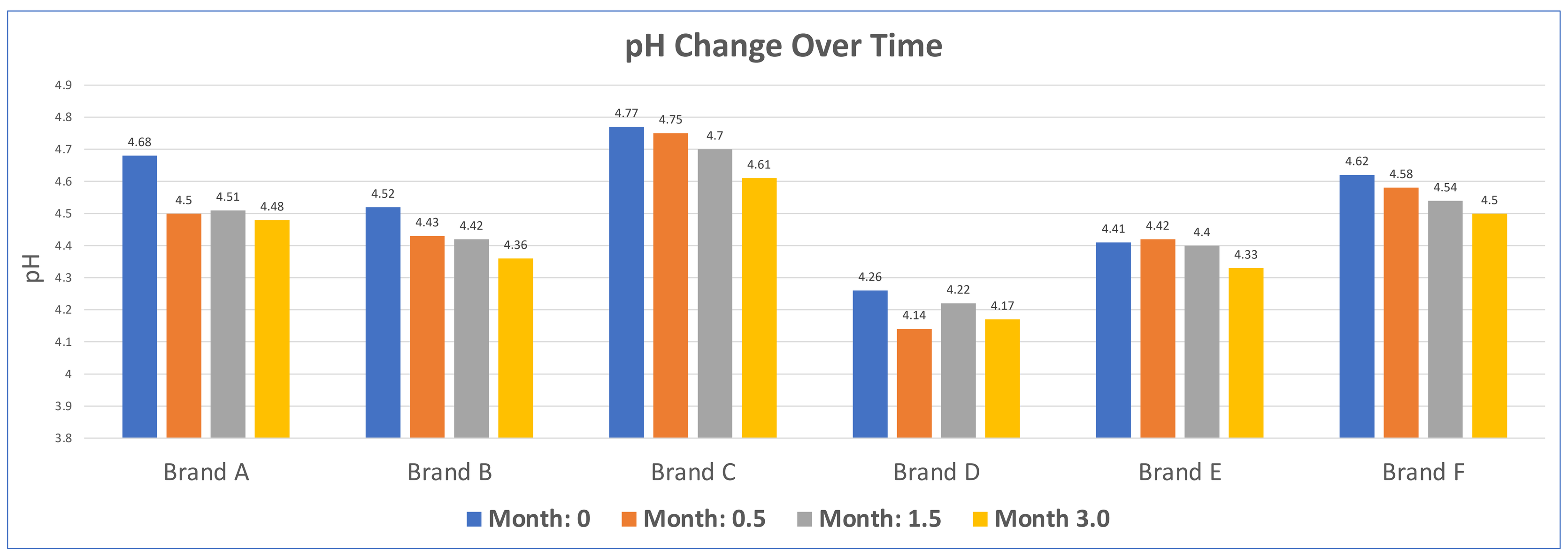

Figure 9: The change in pH over time decreased at some capacity for each brand.

Particle size distributions were not performed on the altered (pH changed, centrifuged, filtered) beer samples.

- Haze decreased to varying degrees with the steps of filtration and centrifugation as hypothesized.
- Altering the pH slightly up and down did not have a large effect on the short-term stability of the haze.

Conclusion

Stokes Law explains this data regarding the size of the radius of the particle correlating directly to the velocity of the particle dropping out of solution, but there are a myriad of different factors beyond simply the particle size. In this investigation we have shown the impacts of different particle sizes and distributions on haze in commercially available products. Loss of haze over time was shown to be as much as 75% reduction in NTUs in some samples, compared to around 7% loss in the best performing samples. When applying the ingredients to the brewing conditions and recipe to the fermentation conditions, dry hopping, chill haze treatment, and additions altering haze, there are a plethora of investigations that could proceed from this. The next stage would be to identify what the particles and proteins are specifically at each timepoint of the investigation.

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