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Carbonation of beer in microbreweries using a bulk gas-to-atomised liquid (BGAL) approach

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Abstract

Why was the work done: In microbreweries, forced carbonation in a bright beer tank using a carbonation stone may require up to 72 hours to reach specification for dissolved carbon dioxide. The mass transfer kinetics in bubbled systems are generally poor due to the small surface area-to-volume ratio in the liquid phase. Alternatively, droplets of beer have a comparatively large surface area-to-volume ratio and can absorb gas at greater rates.
How was the work done: A bulk gas-to-atomised liquid (BGAL) process was evaluated for beer carbonation. Using this method, a spray of beer droplets is carbonated in a closed vessel pressurised with carbon dioxide gas and equipped with a nozzle to atomise the beer. Following testing at prototype scale (3 x 19 L), the approach was evaluated at a pilot scale by retrofitting a 240 L bright beer tank to accommodate a nozzle, and a laboratory brewed IPA was carbonated at a rate of 3.0 L/min.
What are the main findings: The rate of carbonation achieved by the BGAL system was between 3 and 50 times greater than conventional carbonation, the precise improvement was dependent upon the operating conditions for the conventional method. Further, analysis of the finished beer revealed that hop oil compounds in BGAL carbonated beer were statistically similar to the concentration in beer carbonated by conventional means.
Why is the work important: These results suggest that the BGAL system could enhance the carbonation step in microbreweries that use a carbonation stone in bright beer tank, while maintaining the aromatic characteristics of the beer.

Keywords

carbonation; process intensification; bulk gas-to-atomised liquid; hop oil compounds.

Introduction

Carbonation is essential to all aspects of the sensory experience of beer, including (i) imparting a tingling 'bite', (ii) influencing the formation and stability of foam, (iii) facilitating the release of aromatic compounds, and (iv) enhancing mouthfeel through bubble nucleation (Rohner and Tompkins 1970; Briggs et al. 2004; Mosher and Trantham 2021b). Accordingly, achieving the desired level of carbonation for each style of beer is critical to the specification of the brand.

How effectively carbon dioxide gas is absorbed into liquid depends on factors including temperature and vessel pressure (Briggs et al. 2004; Mosher and Trantham 2021b). The solubility of the gas is directly influenced by the liquid temperature, and the vessel pressure is directly proportional to the concentration of CO_2 gas dissolved at the saturation point. These are effectively captured by Henry's law:

$$C_{sat} = \frac{P_{CO_2}}{H_V^{PC}} \qquad \text{(Eq. 1)}$$

where P_{CO2} is the partial pressure of CO_2 gas in the headspace above the liquid, C_{sat} is the saturation concentration, and H_V^{PC} is Henry's law volatility constant in units of *(kPa-L)/g* (Sander et al. 2022). Moreover, the temperature dependence of Henry's law volatility constant can be expressed through the Van't Hoff equation:

$$H_V^{PC}(T) = H_{V,ref}^{PC} \exp\left[\frac{-\Delta H_{sol}}{R}\left(\frac{1}{T} - \frac{1}{T_{ref}}\right)\right] \qquad \text{(Eq. 2)}$$

where $H_{V,ref}^{PC}$ is a reference value of Henry's law volatility constant, ΔH_{sol} is the standard molar enthalpy of solvation, R is the ideal gas constant, and T and T_{ref} are the operating and reference temperatures, respectively (Speers and MacIntosh 2013; Sander et al. 2022). These relationships indicate that the Henry's law volatility constant increases with increasing temperature, which lowers the saturation concentration at the same vessel pressure. Conversely, increasing vessel pressure will increase the saturation concentration at the same liquid temperature.

Although reference to Henry's law volatility constant in beer is limited, a study by Liger-Belair and Cilindre (2021) found that the experimental values of Henry's law volatility constant for water and beer were approximately (within about 10%) equal for carbon dioxide. This corresponds to an absolute difference in predicted saturation concentration of approximately 0.1g/L, or 0.05 vol/vol, at the same pressure. This difference can be attributed to the presence of ethanol and sugars in the beer (Liger-Belair and Cilindre 2021). Accordingly, the computations in this work utilise Henry's law volatility constant for water as a reasonable estimate.

According to standard beer carbonation charts, many (but not all) beer styles have carbonation levels between 2.2 and 2.6 volumes of CO_2 per volume of beer (vol/vol), where one vol/vol of CO_2 is equivalent to a concentration of 1.95 g/L (*https://d163axztg8am2h.cloudfront.net/static/doc/27/24/1ad79390fcfb96639d8136290a17.pdf*; Song et al. 2020; Mosher and Trantham 2021b). However, requirements for carbonation, range from 1.5 vol/vol in some dark beer styles up to 4.0 vol/vol in some wheat beers (Song et al. 2020). Thus, the carbonation process must be sufficiently flexible to meet the requirements of different beer styles.

Beer leaving fermenter contains around 0.75-1.1 vol/vol of dissolved CO_2, and requires carbonation to meet the brand specification (Colby 2020). In many microbreweries, beer is transferred to bright beer tanks where it is subject to forced carbonation with the injection of CO_2 bubbles into the liquid (Munroe 2006; Gomez and Ambrose 2019; Mosher and Trantham 2021b). Most commonly, gas bubbles are sparged into the liquid using a carbonation stone located at the bottom of the bright beer tank. As the bubbles travel through the liquid, they gradually dissolve, with smaller bubbles with a high surface area-to-volume ratio preferred to increase the rate of dissolution. However, achieving small bubbles requires a low volumetric flowrate of carbon dioxide, which can increase the time required to reach the carbonation level (Briggs et al. 2004; Munroe 2006; Steen 2007). Accordingly, a compromise is necessary between the efficiency of gas absorption and carbonation rate.

Carbonation methods in microbreweries using bright beer tanks are variable and depend largely on the preference of the brewer. In breweries requiring a fast turnover, brewers may opt to operate their bright beer tank at high pressure or with a high gas flowrate through the carbonation stone. While this approach may reduce the time for carbonation to 6 to 12 hours, large bubbles will not dissolve fully in the liquid, leading to losses of carbon dioxide, pressure buildup, flavour stripping, undesirable foaming, and a risk of over carbonation. Alternatively, other brewers may set their carbonation stone to operate at a pressure only slightly higher than the target equilibrium pressure, requiring - depending on the style of beer - between 24-72 hours to achieve carbonation. Using method, dark beer styles may require less than 24 hours in a bright beer tank, whereas highly carbonated wheat beers may require up to five days to reach the desired carbonation (Colby 2020; Mosher and Trantham 2021b). Given the large variability of carbonation time required, this work uses 72 hours as an average for the residence time of beer while also considering the sensitivity of results when the residence time ranges from 6 to 96 hours.

The carbonation rate (CO_2) is determined through the gas-to-liquid mass transfer equation:

$$\frac{dC}{dt} = \dot{CO_2} = k_L a(C_{sat} - C(t)) \qquad \text{(Eq. 3)}$$

where C_{sat} is determined by Henry's law (Eq. 1), and $C(t)$ is the measured concentration of CO_2 in the beer. This is measured as volumes of gas at standard temperature and pressure (STP) dissolved in a volume of beer (vol/vol). This quantity can be estimated based on the equilibrium concentration of dissolved CO_2 at the tank operating temperature and pressure, which can be determined from standard carbonation tables, or measured (Taprite Beer Carbonation Tester, 2701-BCT) (*https://d163axztg8am2h.cloudfront.net/static/doc/58/12/a16ec96a075efffb164eaeb83f62.pdf*).

Integrating Eq. 3, the steady-state mass transfer coefficient, $k_L a$, can be calculated by:

$$k_L a = \frac{1}{\tau} * \ln\left(\frac{C_{sat} - C_{in}}{C_{sat} - C_{out}}\right) \qquad \text{(Eq. 4)}$$

where τ is the residence time of droplets in the system, and C_{in} and C_{out} are the CO_2 concentrations in the liquid going in and out of the system. Because τ cannot be easily measured, it is estimated by:

$$\tau = \frac{V_L}{Q} \qquad \text{(Eq. 5)}$$

where V_L is the liquid holdup volume, or the volume of liquid droplets suspended in the vessel at an instant of time, and Q is the liquid flowrate (Sathish et al. 2019).

The conventional approach of bubbling CO_2 into the bulk liquid is characterised as 'atomised gas-to-bulk liquid', where small bubbles of gas are contacted with a large volume of liquid. Although the small bubbles dissolve relatively quickly (given their high surface area-to-volume ratio), the bulk liquid is slowly saturated with gas. This is because of the limit on the volumetric flow rate of gas through the liquid system to prevent the formation of large bubbles with small surface area-to-volume ratios. In industrial fine bubble columns without agitation and a similar hydrodynamic profile to that anticipated in a bright beer tank with a carbonation stone, the $k_L a$ (mass transfer coefficient) is in the range of 0.002-0.05 s^{-1}, depending upon system configuration and bubble size (Heijnen and Van't Riet 1984; Munasinghe and Khanal 2010).

This approach - bulk gas-to-atomised liquid (BGAL) carbonation - uses an inverted hydrodynamic profile to achieve mass transfer (Sathish et al. 2019) where small droplets of liquid are contacted with a large volume of gas. In this system, the liquid phase has a large surface area-to-volume ratio, and each individual droplet becomes saturated with gas more quickly than is possible for a large volume of liquid. Further, a high liquid throughput is preferred to enhance the atomisation of the liquid to smaller droplets with a $k_L a$ up to 100 times greater than that of bubble columns, indicating more effective mass transfer (Sathish et al. 2019).

The carbonation rate in any system can be maximised by increasing $k_L a$ and the driving force term (C_{sat}-$C(t)$). Both parameters are dependent on the operating conditions and the system configuration,

including liquid temperature, vessel operating pressure, droplet or bubble size, and liquid or vapour holdup. Although the carbonation rate in a conventional system will decrease over time as the concentration of dissolved CO_2 increases, reducing the magnitude of the driving force, the BGAL system (a steady state in-line procedure) offers a consistent carbonation rate throughout the process.

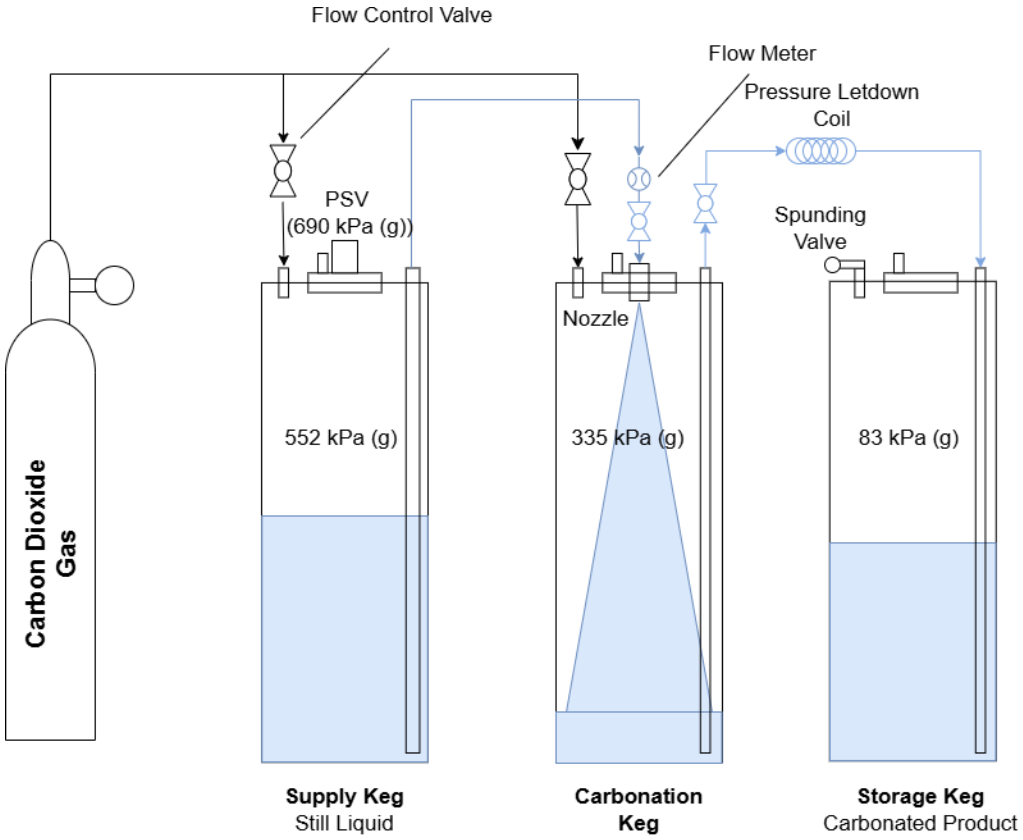
This work considers the rate of carbonation using the BGAL approach and associated mass transfer coefficient in the system. The efficacy of the BGAL approach was evaluated in a prototype system using three 19 L Cornelius kegs connected in series. With promising results from the prototype, the BGAL approach was evaluated in a 240 L pilot system. This enabled the comparison of BGAL over the use of carbonation stone in a bright beer tank under microbrewery operating conditions and scale.

Methods and materials

Evaluation of the BGAL approach at the prototype scale

The prototype system was used to evaluate the efficacy of the bulk gas-to-atomised liquid (BGAL) approach prior to pilot testing. Although the theoretical basis for utilising droplets to enhance gas-to-liquid mass transfer was sound, it was unclear if additional considerations specific to the beer-gas matrix, such as liquid composition and complexity, would make the approach impractical. This system (Figure 1) consisted of three 19 L Cornelius kegs connected in series. For experiments, the kegs were purged and pressurised with CO_2 to allow for pressure-driven flow. The leading keg or 'supply keg', contained chilled water or beer (with 0.88 vol/vol of residual CO_2 from fermentation at 20°C), and was equipped with a 690 kPa (g) (kilopascal-gauge) pressure safety valve (PSV) (Colby 2020). The middle keg or 'carbonation keg', was equipped with a modified lid that accommodated a spray nozzle in the centre (BETE Fog Nozzle, Inc., WL-4 60) such that liquid entered the carbonation keg as droplets falling through an atmosphere of CO_2. The carbonated product flowed from the bottom of the carbonation keg to the third keg ('storage keg') maintained at the target pressure with a 'spunding' valve that released displaced gas to the atmosphere as the keg was filled with liquid.

Figure 1. Prototype system configuration (patent pending)



$$Tubing\ Length\ (m) = \frac{Carbonation\ Keg\ Pressure\ (kPa\ (g)) - Storage\ keg\ Pressure(kPa\ (g))}{Tubing\ Resistance\left(\frac{kPa\ (g)}{m}\right)} \quad \text{(Eq. 6)}$$

To avoid beer foaming in the storage keg, a 'pressure letdown coil' was installed ahead of it to assure a gradual pressure drop between the carbonation and storage kegs. The length of tubing to achieve the desired pressure drop in the coil was estimated (Eq. 6) where tubing resistance is a function of the materials of construction and diameter of the tubing.

Liquid holdup in the prototype system

Knowledge of liquid holdup, V_L, which is the steady state volume of liquid droplets present in an enclosed space (the 'carbonation keg'), is required to calculate k_La (Eq. 4 and 5). The nature of the BGAL prototype configuration precluded direct measurement of the volume of liquid in the carbonation keg during normal operation as detailed previously. Therefore, the following approach was used to approximate the volume at different flowrates.

The modified lid with spray nozzle, was held above a floor drain at a height of 0.6 m, the same height as the Cornelius kegs. The flow control valve was opened, taking less than a second for the spray to reach the floor. The instantaneous liquid holdup volume could therefore be measured by rapidly opening and closing the flow control valve and collecting the liquid, which was equivalent to the volume of liquid contained in the spray cone within the height of the Cornelius keg.

Using this methodology, the liquid holdup experiments were initiated by filling the supply keg with water and pressurising it with CO_2 to approximately 550 kPa (g), an arbitrary pressure chosen to provide adequate driving force for the range of liquid flowrates. A flowmeter with an integrated flow control valve (King Instrument 7530) in the line between the supply keg and carbonation keg was used to set the flowrate of the liquid through the nozzle. A manual ball valve installed between the flowmeter and the nozzle was used to start and stop the flow. By starting and stopping the flow for prescribed periods of time and collecting the intermittent flow in individually tared 200 mL sample containers, liquid holdup volume could be determined as a function of flowrate.

Twenty holdup samples were collected for each of four liquid flowrates between 4 and 6 L/min. The volume of each sample was determined by weighing the final mass of liquid and converting to volume using the standard density of water.

Mass transfer coefficient measurements in the prototype system

To determine k_La in the prototype system and understand its relationship to operating conditions, a series of experiments were performed under various carbonation vessel gas pressures. Initially, all three kegs were purged with CO_2 followed by pressurisation with CO_2 to levels specified in Table 1. A floor scale was placed under both the supply and carbonation kegs and tared, allowing for observation of the approximate liquid level within each keg. An experiment was initiated by opening the ball valve between the supply and carbonation kegs, which allowed the liquid from the supply keg to spray into the carbonation keg. Once a small amount of carbonated product (about 1.8 kg) had pooled in the base of the carbonation keg, the ball valve between the carbonation and storage kegs was opened and carbonated product began to accumulate in the storage keg. Due to the nature of the pressure driven flow, it was difficult to achieve a perfect steady state. Therefore, the ball valves upstream and downstream of the carbonation keg were toggled on and off to ensure that neither too much nor too little liquid collected in the bottom of carbonation keg, leading to either insufficient droplet residence time or cavitation in the pressure drop coil, respectively. This volume was maintained between 1.8- 3.7 L, corresponding to an approximate mass of 1.8-3.7 kg as monitored by the floor scales.

Table 1. Vessel pressures to determine system k_La

Supply keg (kPa (g))	Carbonation keg (kPa (g))	Storage keg (kPa (g))
483	276	83
552	345	83
620	414	83

To reach a target flowrate of 4 L/min through the system and produce acceptably small droplets, the pressure drop across the nozzle was maintained at approximately 206 kPa (g), a set point determined by observation of the spray pattern in open air under different CO_2 pressure scenarios. The storage keg pressure was set to 83 kPa (g), typical of the storage pressure for kegged beer. Given these requirements, three sets of operating conditions (Table 1) were investigated to determine the relationship between k_La and pressure in the carbonation keg.

Two trials were run for each set of operating conditions. For each trial, approximately 8 L of water was carbonated and collected in the storage vessel. The final concentration of CO_2 was measured in triplicate using a Taprite Beer Carbonation Tester (2701-BCT). The mass transfer coefficient, k_La, was calculated using Eq. 4. Using the same method, laboratory-produced American lager with an ABV of 4.9% was carbonated. For calculation of k_La, the beer in the supply keg was assumed to contain 0.88 vol CO_2/vol beer, the equilibrium pressure for fermentation at approximately 20°C and atmospheric pressure (Colby 2020).

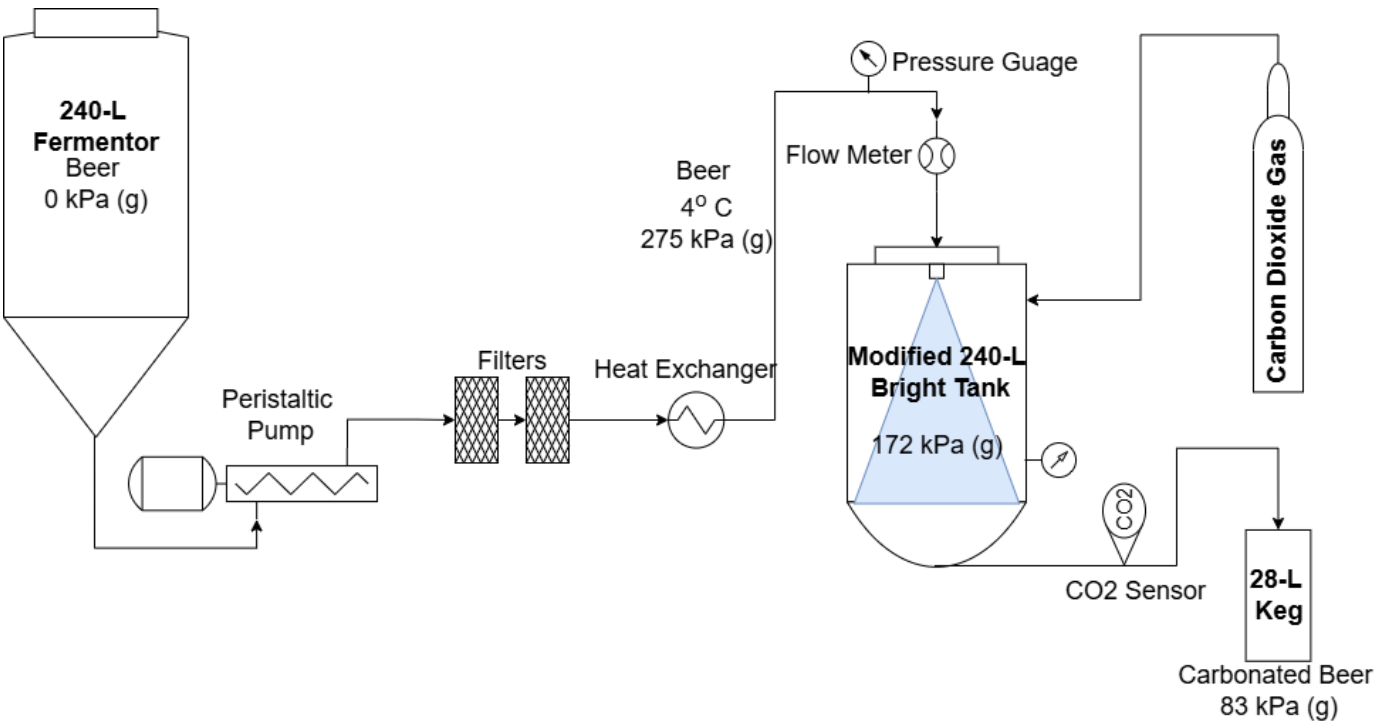
The BGAL approach at pilot scale

The pilot system for BGAL carbonation (Figure 2), utilised a retrofitted bright beer tank as the carbonation vessel. The tank was fitted with a pressure safety valve (PSV) set to 207 kPa (g) and a McMaster-Carr 303 Stainless Steel Fogging Nozzle was installed in the centre of the lid. Tests were initiated by purging and pressurising the bright tanks with CO_2 at 172 kPa (g). The beer was pumped from the fermenter through 5 micron (Aquaboon) and 1 micron (Aquaboon) inline filters and the temperature reduced below 4°C using a plate heat exchanger (Blichmann Engineering). The tank was continuously supplied with CO_2 to maintain the required carbonation vessel pressure. The carbonated beer pooling at the base of the tank was transported to 28L storage kegs equipped with spunding valves set at the target storage pressure.

Liquid holdup in the pilot system

Liquid holdup experiments were conducted with water using the modified method of Sathish et al. (2019). The experimental apparatus is shown in Figure 3. The tank was filled with a known volume of water (V_{system}) selected to ensure that no water pooled in the base of the carbonation vessel during steady state operation. The outlet of the tank was

Figure 2. Pilot system configuration (patent pending)



attached to a short hose connected to a peristaltic pump, which pumped water through a pressure gauge, flowmeter, and the nozzle at the top of the carbonation vessel. For each experiment, the pump was turned on, and the system achieved steady state as confirmed by constant line pressure and liquid flow readings.

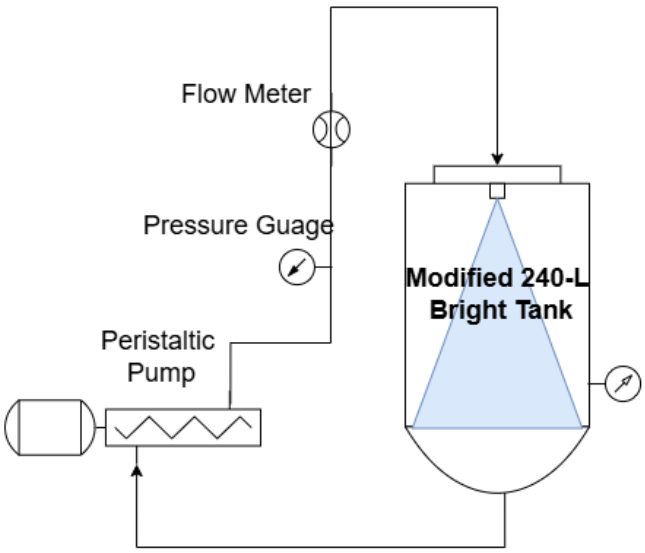
The holdup volume, V_L was measured by collecting all water in the tubing upstream and downstream of the carbonation vessel, V_{tubing} and subtracting that measured volume from the volume supplied to the system:

$$V_L = V_{system} - V_{tubing} \qquad \text{(Eq. 7)}$$

Additionally, as a check on mass closure, the volume of water between the two shutoff valves was measured. Slight mass losses were anticipated due to liquid remaining in the vessel from wall coating and losses during the collection process, mass closure was between 98-100%. The experiments were carried out, in triplicate, at flowrates of 4.7 L/min, 5.7 L/min, and 6.6 L/min, corresponding to a nozzle pressure drop of 137 kPa (g), 172 kPa (g), and 207 kPa (g), respectively. This enabled the relationship between volumetric flowrate and liquid holdup volume to be determined.

The quantification of V_L may appear trivial, but its measurement and accuracy are important to accurately determine k_La. Further, V_L for industrial spray systems is often overlooked or poorly estimated due to hydrodynamic complexities in the system.

Figure 3. Evaluation of liquid holdup in the pilot system



While the proposed method allows for high resolution measurements with an error of ± 8%, the previous method (Ercan 1989) for quantification of V_L resulted in an error of ± 40%.

Measurement of the mass transfer coefficient in the pilot system

Eq. 4 was used to calculate k_La for the pilot system. The beer (IPA) in the pilot scale trials was fermented (240 L) at 18°C for five days before reducing the temperature to 7°C for dry hopping. The beer was allowed to increase to a pressure of 69 kPa (g) until fermentation was 80% complete. After a further four days, the temperature was reduced to -1°C, dropping yeast and hop material which was removed. The carbon dioxide concentration of the fermented beer was 2.13 ± 0.07 vol/vol (Taprite Beer Carbonation Tester). Although the CO_2 level was near the specification, additional carbonation was required.

All lines and equipment were sanitised by cleaning in place (CIP) using the fogging nozzle, the retrofitted tank was purged with CO_2, pressurised to 172 kPa (g), and a glycol chiller was set to 4°C. The peristaltic pump was adjusted to maintain a line pressure between 248 kPa (g) and 275 kPa (g), corresponding to a nozzle pressure drop of 69-103 kPa (g) and a liquid flowrate of approximately 3.0 L/min. On-line data included temperature and pressure at the inlet and outlet and CO_2 concentration at the outlet, measured in real-time with an Anton Parr Carbo 5100 dissolved CO_2 sensor. All data were recorded at 30 second intervals. Once carbonated, the beer was stored in four 28L storage kegs for further analysis. The remaining beer (approximately 120 L) was carbonated using the conventional method of bubbling CO_2 using a carbonation stone (Blichmann Engineering) set to 103 kPa (g) and 0°C. This method of carbonation required six days to reach a target CO_2 concentration of 2.7 vol/vol.

Flavour and aroma compounds in beer from BGAL and conventional carbonation

The operation of conventional bright beer tanks are configured to limit the loss of desirable flavour and aroma compounds (Mosher and Trantham 2021b). Many of the aromatic compounds in beer are attributed to volatiles derived from hop oils, including hydrocarbons, oxygenated compounds

and sulphur containing compounds (Dietz et al. 2020). Due to the volatility of these compounds, there was concern that atomisation of the beer would cause their release due to the high surface area-to-volume ratio of the droplets.

To ensure that the composition of the beer was maintained during atomisation, the lager carbonated in the prototype system was analysed by Qualitative GC-MS and compared to the GC-MS spectrum of the same batch of beer carbonated conventionally. Analysis was performed using an Agilent 8890 GC System coupled with an Agilent 5988B GC/MSD with an inlet temperature of 260°C and a MS temperature of 280°C. Agilent MassHunter® Qualitative Analysis was used to analyse both chromatograms and identify peak spectra using the NIST20 database. Following this, a secondary analysis was conducted targeting hop compounds at low concentration.

To determine if there were any differences in the concentration of hop compounds retained by BGAL carbonation and conventional carbonation, two samples of the BGAL-carbonated IPA were bottled from each of the four 28L storage kegs. The hop concentration was measured in uncarbonated IPA bottled directly from fermenter and in conventionally carbonated IPA were bottled at the end of carbonation in the bright beer tank.

Headspace solid-phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC-MS) was used to analyse myrcene, linalool, and geraniol in all the IPA samples. This was performed using an Agilent 5975C GC/MSD coupled with an Agilent 7890 GC. The SPME fibres were 50/30 µm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) (Cai et al. 2017). The fibres were conditioned according to the manufacturer instructions by inserting them in the desorption unit of the Combi PAL autosampler (Supelco, PA, USA).

Samples were brought to room temperature and then centrifuged to remove hop particulates. An aliquot (4 mL) was placed into a 10 mL amber glass vial with 2g NaCl. The fibres were automatically introduced through the capped septum and exposed to the headspace of the vial in a thermostatic bath at 50°C for 10 min. The fibres were then removed from the sample vial and inserted into the heated injector of the GC-MS (260°C) with a 120 s desorption time (Cai et al. 2017).

The volatile compounds were separated on a tandem column configuration to achieve a mid-polarity separation; the first polar column was a DB-Wax column (30 m x 0.530 mm x 0.50 µm). The second non-polar column was a DB-5 column (30m x 0.530 mm x 0.50 µm), both purchased from Agilent (Santa Clara, California, USA). After injection, the column temperature was held at 40°C for 3 min, increased to 220°C at 7°C/min, and then held at 220°C for 11.29 min. The mass spectrometer was operated in electron ionisation (EI) mode (70 eV) ion source and MS quadrupole held at 230°C and 150°C, respectively, with mass-to-charge ratio values recorded over a range of 34 to 450 m/z.

Quantification was using a Agilent MassHunter® Workstation (Agilent, Santa Clara, CA, USA) with confirmation identification using NIST11 mass spectral database and BenchTop/PBM with Wiley Registry of mass spectral data (7th edition, Palisade Corporation, New York, USA). Assay controls were analysed and found to be +/- 10% of assay control value with 6.2% inter-assay CV and 7.9% intra-assay CV. All standards were purchased from Fischer Scientific (Hampton, NH, USA) with the highest possible purity.

Results and discussion

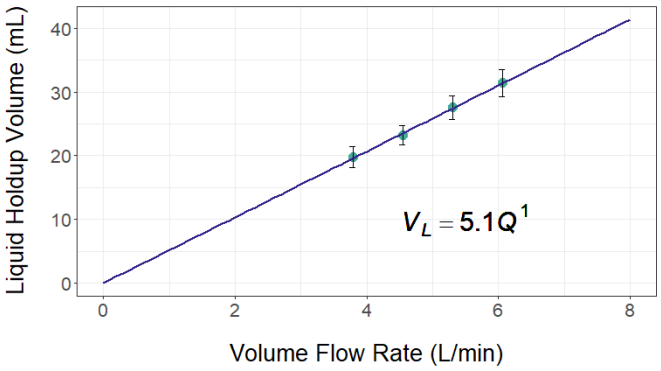
Prototype system

To determine the relationship between volumetric flowrate and liquid holdup volume (V_L) in the prototype system, 20 samples were taken at each flowrate. Figure 4 shows the linear relationship between liquid holdup and volumetric flowrate in the prototype system. The equation of the fit is used to calculate V_L based on the recorded volumetric flowrate in carbonation tests. This relationship was used to calculate k_La using Eq. 4.

Following determination of V_L, carbonation tests were carried out in the prototype system to determine the volumes of dissolved CO_2 per volume of liquid (vol/vol) as a function of carbonation keg pressure for the operational setpoints (Table 1). Experiments were carried out using both water and American lager. Table 2 summarises the results

of eight carbonation trials in the prototype system, including carbonation keg pressure, liquid flowrate (Q), liquid holdup volume (V_L), the CO_2 saturation concentration calculated from Henry's law (C_{sat}), the measured CO_2 concentration in the carbonated liquid (C_{out}), and the mass transfer coefficient (k_La).

Figure 4. BGAL prototype liquid holdup (V_L) relationship to liquid volumetric flowrate (Q)



The carbonation keg pressures for the trials using the American lager were based on the results with water. As carbonation keg pressures of 335 kPa (g) and 414 kPa (g) carbonated water to the desired levels, the same conditions were used for carbonating the American lager. Both keg pressures carbonated the lager to 2.3 vol/vol, within the style specification. Similar trends in k_La are observed for trials 7 and 8 using the American lager. However, the calculated values are reduced due to the presence of residual CO_2 in the fermented lager.

Figure 5 shows the final vol CO_2/vol beer in each of the eight carbonation trials, superimposed on a standard saturation pressure vs. temperature chart used in the brewing industry. These results show the final concentration of CO_2 in water increasing with carbonation keg pressure, highlighting the importance of liquid temperature in determining these concentrations. For example, the CO_2 concentration measured in Trials 3 and 5 are almost identical. The liquid in Trial 3 was approximately 1.5°C colder than in Trial 5, and the CO_2 was more readily dissolved despite the 69 kPa difference in carbonation keg pressures. These tests suggest that beer can effectively be carbonated by the BGAL method, with scale up to 240L to evaluate at microbrewery scale.

Pilot system

Experiments were conducted on the pilot system to determine V_L and k_La for BGAL carbonation at a practical brewery scale. Liquid holdup (V_L) as a function of nozzle pressure drop is reported in Figure 6. This derived correlation is utilised in computations of k_La using Eq. 4 and 5. During these liquid holdup experiments, both liquid flowrate (Q) and nozzle pressure drop (ΔP) were monitored, allowing for derivation of a relationship between the two parameters:

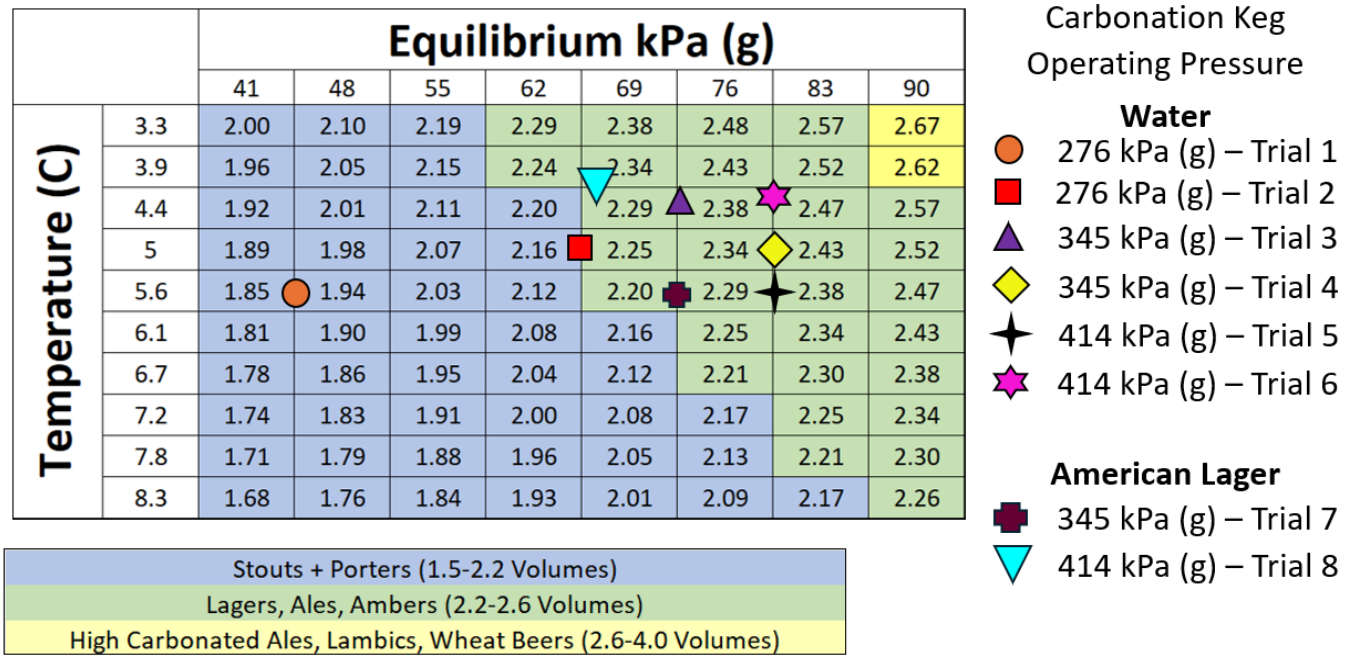
$$Q\left(\frac{L}{min}\right) = 0.03 * \Delta P(kPa) \pm 0.1\frac{L}{min} \qquad \text{(Eq. 8)}$$

Because the pressure gauge offered a greater level of precision than the flowmeter, this correlation was used to determine flowrate during the subsequent carbonation experiments.

Table 2. BGAL prototype carbonation

Trial	Pressure (kPa (g))	Q (L/min)	V_L (L)	T (°C)	C_{sat} (vol/vol)	C_{out} (vol/vol)	k_La (s^{-1})
1	276	3.8 ± 1.9	0.02 ± 0.001	5.6 ± 0.0	5.1 ± 0.0	1.9 ± 0.05	1.5 ± 0.7
2	276	5.3 ± 1.9	0.03 ± 0.002	5.0 ± 0.6	5.3 ± 0.1	2.2 ± 0.09	1.7 ± 0.6
3	335	4.5 ± 1.9	0.02 ± 0.002	4.4 ± 0.0	6.4 ± 0.0	2.3 ± 0.05	1.4 ± 0.6
4	335	4.9 ± 1.9	0.03 ± 0.002	5.0 ± 0.6	6.3 ± 0.1	2.3 ± 0.03	1.5 ± 0.6
5	414	4.9 ± 1.9	0.03 ± 0.002	5.6 ± 0.0	7.1 ± 0.0	2.4 ± 0.09	1.3 ± 0.5
6	414	4.5 ± 1.9	0.02 ± 0.002	4.3 ± 0.3	7.4 ± 0.1	2.4 ± 0.04	1.2 ± 0.5
7	335	4.9 ± 1.9	0.03 ± 0.002	5.6 ± 0.0	6.1 ± 0.0	2.3 ± 0.09	1.0 ± 0.4
8	414	4.5 ± 1.9	0.02 ± 0.002	4.4 ± 0.0	7.4 ± 0.0	2.3 ± 0.00	0.8 ± 0.3

Figure 5. Concentration of CO_2 (vol/vol) in the prototype system carbonation tests



From *https://d163axztg8am2h.cloudfront.net/static/doc/27/24/1ad79390fcfb96639d8136290a17.pdf.*

The mass transfer coefficient (k_La) was calculated for each of the four kegs of IPA carbonated in the bright beer tank maintained at 172 kPa (g) (Table 3). The variations observed in the final CO_2 concentration (C_{out}) across the kegs can be attributed to the changing system temperature throughout the experiments. The average system temperature while filling Keg 1 was 3°C warmer than for the remaining kegs as the hoses and auxiliary equipment were at room temperature. Indeed, the system temperature did not achieve steady state until halfway through filling Keg 2, where it remained steady until the operation was paused briefly to ensure Keg 4 was precisely filled, and the system temperature increased slightly. These observations illustrate that precise control of the system temperature is important if the BGAL system was used to carbonate different styles of beer.

The k_La in the pilot system was approximately 10 fold less (Table 3) than in the prototype system due to the reduced operating pressure and the greater initial dissolved CO_2 ex-fermenter, which reduced the magnitude of the logarithmic term in Eq.4. However, the magnitude of k_La for the pilot BGAL system was up to 100 times greater than that in bubble systems (0.002-0.05 s^{-1}) (Heijnen and Van't Riet 1984; Munasinghe and Khanal 2010). Furthermore, the data show that precise control of the beer temperature, flowrate, and carbonation vessel pressure would allow the efficient carbonation of different styles of beer utilising a BGAL approach. These results show that beer was effectively carbonated to desired gas volumes at a volumetric beer flowrate that exceeded 132 L/h through the 240 L BGAL system. This is at least a 40-fold rate improvement compared to the 3.4 L/h achievable by conventional forced carbonation, assuming an average 72 hour residence time in a 240 L bright beer tank to reach equilibrium.

Figure 6. BGAL pilot liquid holdup (V_L) relationship to nozzle pressure drop (ΔP)

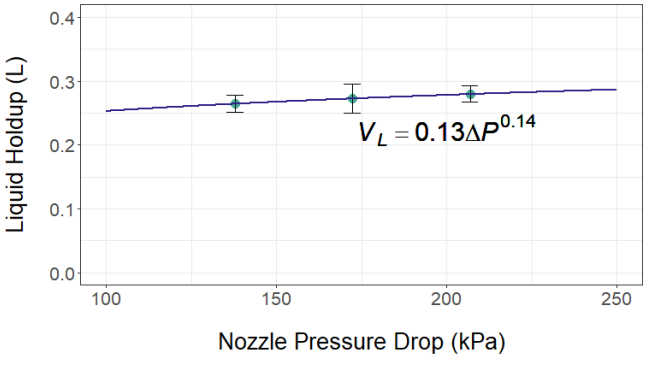


Table 3. BGAL pilot carbonation

Storage Keg	ΔP (kPa)	Q (L/min)	V_L (L)	T (°C)	C_{sat} (vol/vol)	C_{out} (vol/vol)	k_La (s^{-1})
1	93.1 ± 0.0	3.0 ± 0.2	0.25 ± 0.01	9.7 ± 1.8	3.4 ± 0.2	2.4 ± 0.1	0.05 ± 0.03
2	92.6 ± 13.8	3.0 ± 0.6	0.25 ± 0.01	6.8 ± 0.3	3.7 ± 0.04	2.6 ± 0.1	0.07 ± 0.03
3	88.7 ± 7.3	2.9 ± 0.4	0.25 ± 0.01	6.3 ± 0.1	3.7 ± 0.02	2.8 ± 0.0	0.11 ± 0.02
4	66.9 ± 11.7	2.2 ± 0.5	0.24 ± 0.01	6.8 ± 1.1	3.7 ± 0.2	2.7 ± 0.2	0.07 ± 0.04

Due to the large variability of carbonation times during conventional bright beer tank carbonation, it is important to compare these results to a wide range of assumed conditions. As noted previously, carbonation may require from 6 to 96 hours, depending on the operating conditions, beer style, and system configuration. At a scale of 240 L, these timescales correspond to a carbonation rate ranging from 2.5 L/h to 40 L/h. Therefore, even methods to quickly carbonate beer using a high operating pressure in the bright beer tank or a high gas flowrate through the carbonation stone are at least three times slower than the BGAL approach at this scale.

Flavour and aroma compounds in BGAL and conventionally carbonated beer

The comparative qualitative GC-MS chromatograms from the protype and conventionally carbonated American lager are shown in Figure 7. Among the compounds are aldehydes, ketones, and alcohols that are by products of the Maillard reaction during wort boiling (Šmogrovičová and Dömény 1999; Hellwig and Henle 2020; Rudnicki et al. 2025). The similarity of the two spectra suggest that the composition of beer was maintained when atomised in the prototype system, despite the greater surface area-to-volume ratio of the droplets. This is likely as the residence time of the droplets within the BGAL system was not long enough for substantial loss of these compounds. Despite this, targeted quantitative analysis of hop compounds was used with known volatiles found in low concentrations.

Table 4 reports the concentration of myrcene, linalool, and geraniol from four measurements of each beer sample using SPME, with two replicates for each of two bottled samples. These results indicate that BGAL carbonated beer had similar concentrations of linalool and geraniol to to that of conventionally carbonated beer. Although the error in the measurement of myrcene in the bright beer tank is large, the result suggests myrcene

Figure 7. Qualitative GC-MS chromatograms of BGAL and conventionally carbonated American lager

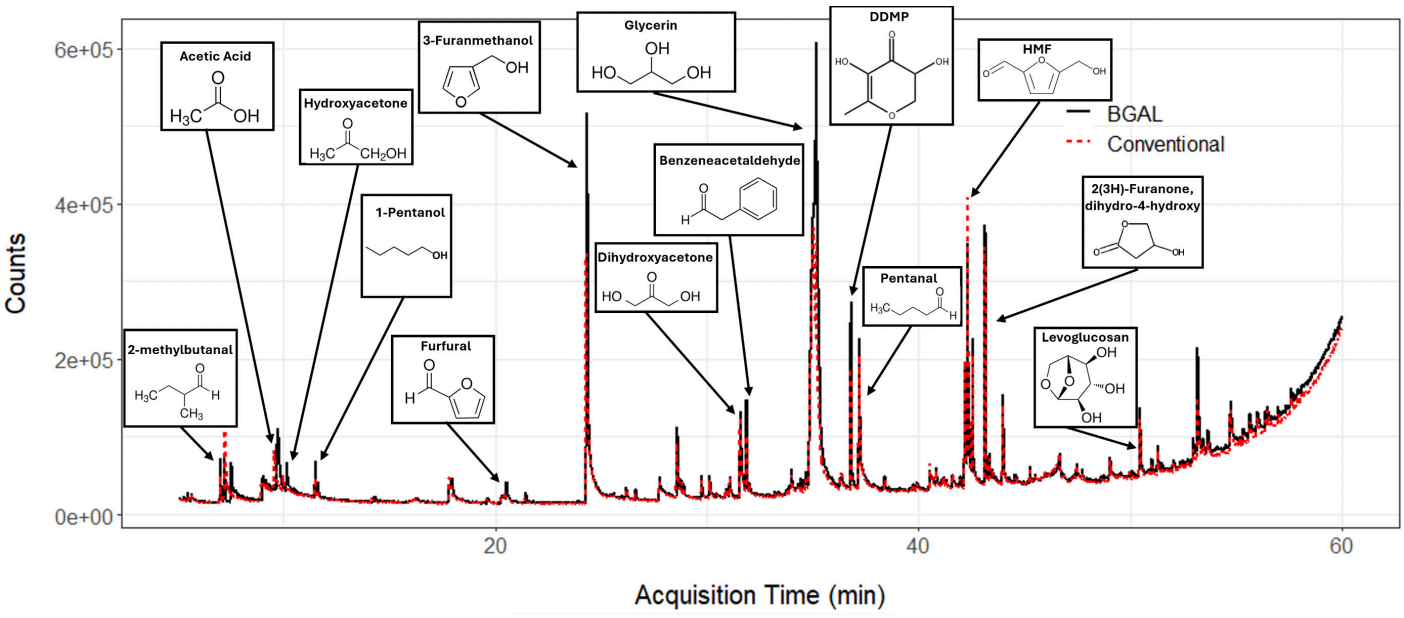


Table 4. Concentration of hop oils in BGAL and conventionally carbonated beers

Sample	Myrcene (µg/kg)	Linalool (µg/kg)	Geraniol (µg/kg)
Fermenter	1872 ± 91	3012 ± 349	1674 ± 112
Keg 1 (BGAL carbonation)	1008 ± 41	2968 ± 46	1488 ± 199
Keg 2 (BGAL carbonation)	1076 ± 166	2863 ± 183	1025 ± 119
Keg 3 (BGAL carbonation)	1440 ± 18	2988 ± 39	926 ± 329
Keg 4 (BGAL carbonation)	698 ± 107	3453 ± 217	825 ± 520
BBT (conventional carbonation)	1996 ± 749	3481 ± 401	1049 ± 424

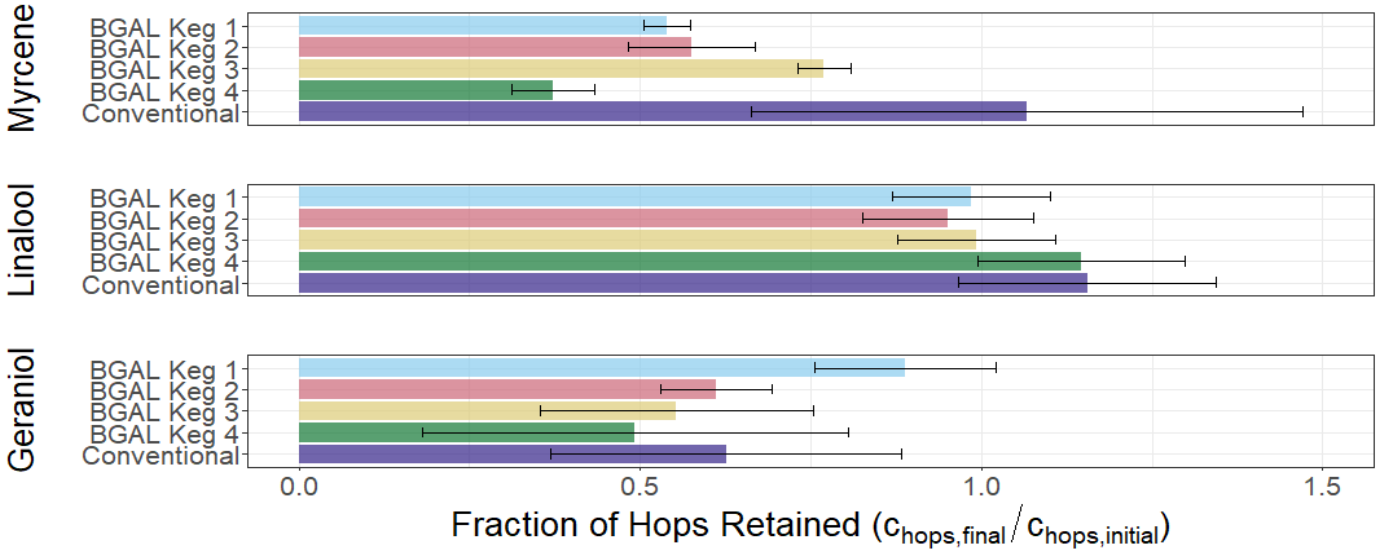
levels are higher in conventionally carbonated beer than in beer carbonated using BGAL.

To compare the volatilisation loss by the two carbonation methods, the fraction of hop oils ($C_{(hops,final)}/C_{(hops,initial)}$) retained in the beer after carbonation are presented in Figure 8, with $C_{initial}$ the concentration of compounds in the beer from fermenter prior to carbonation (Table 4).

Across the three hop compounds, only myrcene shows a statistically significant difference in retention across carbonation methods based on a one-way ANOVA test ($p<0.05$). However, the absolute difference in retention is relatively small given the large variance in the data. The results suggest that conventional carbonation results in volatilisation of up to 40% of the initial myrcene concentration while BGAL carbonation results in volatilisation of 20-60%. Perhaps more importantly, the absolute concentration of myrcene measured in the final BGAL carbonated beer (Table 4) remained between 700 and 1400 µg/kg, above the reported aroma threshold of 350 µg/kg (Klimczak et al. 2023). However, there is no statistical difference (one-way ANOVA test ($p>0.05$)) for the levels of linalool and geraniol retained by conventional carbonation or BGAL carbonation.

Given that the hydrocarbon myrcene is more volatile in beer than the oxygenated compounds linalool and geraniol, these observations support the original hypothesis that highly volatile hop compounds may more rapidly volatilise from droplets of beer than in a conventional carbonation system due to the greater surface area-to-volume ratio. Nevertheless, these results indicate that hop oils are sufficiently maintained using BGAL carbonation such that the aromatic characteristics of the beer will be broadly consistent between the two carbonation methods.

Figure 8. Fraction of hop compounds retained across BGAL and conventional carbonation



Haze, foam, and shear stress

In addition to aroma and flavour, other quality markers in the evaluation of beer include haze together with foam formation and stability. In general, these characteristics are a consequence of upstream processing decisions, from barley malt and yeast, processing temperature, and transfer/recirculation during mash and wort processing (Speers et al. 2003; Stoupis et al. 2003; Briggs et al. 2004; Barnes 2006; Leiper and Miedl 2006; Stewart 2006; Wang and Ye 2021; Mosher and Trantham 2021a, c; Bamforth 2023; Li et al. 2024). Haze formation and stability reflects the interactions between haze active proteins (from malt) and polyphenols (from malt and hops) and the presence of high molecular weight β-glucan (from malt) (Speers et al. 2003; Stewart 2006; Wang and Ye 2021). Similarly, foam stability is dependent upon the interactions between proteins and peptides (from malt) and iso-α acids (from hops) (Stewart 2006; Bamforth 2023; Li et al. 2024).

The processing steps in the production of beer seek to minimise shear stress (Briggs et al. 2004; Barnes 2006; Leiper and Miedl 2006; Mosher and Trantham 2021a, c). Upstream of the fermenter, maintaining low shear stress minimises the release of β-glucan and proteins which affect the formation and stability of haze and foam (Barnes 2006; Leiper and Miedl 2006; Mosher and Trantham 2021b; Wang and Ye 2021). Downstream of the fermenter, shear stress is managed to preserve the quality of yeast for re-pitching (Stoupis et al. 2003; Briggs et al. 2004; Munroe 2006). While conventional carbonation in a bright beer tank is a low-shear process, larger scale breweries utilise the Venturi effect for in-line steady-state carbonation, resulting in higher shear forces to achieve adequate gas mixing (Descoins et al. 2006; Bottani et al. 2013; Mosher and Trantham 2021b). Given that the BGAL method of carbonation would allow smaller scale breweries to apply a similar steady-state continuous carbonation, it is useful to compare the shear forces from the two carbonation methods.

For this analysis, the shear stress experienced by beer in the pilot system was calculated using the Darcy-Weisbach friction factor formula with the Blasius correlation for a smooth pipe:

$$\tau = \frac{f\rho v^2}{8} = \frac{0.32Re^{-\frac{1}{4}}\rho\left(\frac{Q}{A}\right)^2}{8} \qquad \text{(Eq. 9)}$$

where τ is the shear stress, *Re* is the Reynold's number, *ρ* is the beer density, *Q* is the volumetric flowrate of the liquid, and *A* is the cross-sectional area of the venturi or nozzle orifice. Although the specific dimensions of commercial venturi carbonation systems are largely proprietary, technical data from the Bucher Denwel Inline Carbonation Automatic Unit (model DCS500A) was used to model the system design *(https://www.bucherunipektin.com/sites/default/files/2024-04/Inline Carbonation_automatic unit_0.pdf).*

Table 5. Computation of shear stress

	Standard pipe flow	Commercial Venturi carbonator	Pilot scale BGAL carbonator
Flowrate (m^3/s)	0.013	0.013	0.00005
Diameter (m)	0.1	0.03	0.0023
Beer Density (kg/m^3)	1010	1010	1010
Beer Viscosity $(Pa \cdot s)$	0.0015	0.0015	0.0015
Cross-Sectional Area (m^2)	0.008	0.0007	0.000004
Velocity (m/s)	1.6	18.6	12.5
Reynold's Number (unitless)	110,000	380,000	19,000
Friction Factor (unitless)	0.018	.013	0.027
Shear Stress (Pa)	5	570	530

A standard β value of 0.3 was chosen, where $\beta = d_{throat}/D_{pipe}$ to determine the cross-sectional area of the venturi (Descoins et al. 2006; Bottani et al. 2013). The appropriate values used for computation are provided in Table 5. This analysis indicates that the shear stress experienced by the beer as it travels through the nozzle (approximately 530 Pa) is comparable with the shear stress in a commercial venturi carbonator (approximately 570 Pa). Jaspe and Hagen (2006) reported that protein unfolding required large shear rates (> 20,000 Pa), in low viscosity media such as beer. While minor conformational transitions which theoretically could disrupt the stability of haze and foam active proteins are possible at shear rates as low as 3 Pa, the shear stress (5 Pa) computed for typical pipe flow would sufficient to cause these reorganisations (Jaspe and Hagen 2006). Lange et al. (2001) reported a decline in yeast cell viability above 1300 Pa, but they observed no evidence of cell lysis at shear rates up to 2800 Pa. Accordingly, it is unlikely that the shear rates from beer flow through the nozzle are sufficient to disrupt the structures of haze and foam active proteins or cause yeast cell lysis.

Conclusions

The work reported here evaluated a bulk gas-to-atomised liquid (BGAL) approach to enhance carbonation in microbreweries that utilise conventional carbonation in a bright beer tank using a carbonation stone. Tests using the 240 L pilot scale system showed that a bright beer tank could be retrofitted to operate in a BGAL configuration and carbonate beer an average of 40 times faster than conventional bright tank carbonation. Further, qualitative GC-MS analysis of carbonated American lager coupled with the SPME analysis of hop oil retention across different carbonation methods indicated that compounds were maintained in the beer during BGAL carbonation. However, as these analyses were not comprehensive, further evaluation of BGAL carbonation should include a fuller quantitative analysis of compounds and sensory analysis of key aroma characteristics. Similarly, although analysis of shear stress shows comparable values to flow through an in-line venturi carbonator such that haze and foam stability are unlikely to be disrupted, these characteristics require further analysis to ensure consistency across carbonation methods.

Although these results are supportive of the application of BGAL in microbreweries, future investigations should include a technoeconomic analysis of the system to determine potential cost savings. A life cycle assessment could also yield useful insight as brewers seek to reduce their environmental footprint. Given the reduced time for carbonation using the BGAL approach, it is likely that energy use, operating costs, and emissions associated with the beer carbonation would be reduced. Further, the different rates of volatilisation of hop compounds pose questions about the intermolecular interactions of compounds, particularly the preferential retention of hop compounds with similar molecular weight and composition. In conclusion, this investigation into the utilisation of a BGAL system in microbreweries suggests the approach could be used to intensify beer carbonation while preserving the aromatic qualities of the beer.

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Conflict of interest

Alexandra Jean, Jordan Funkhouser, and Robert Brown are the inventors of a pending patent application relating to this work (US20250325946, Device for Rapid Carbonation of Beverages; Application No 19184904). The authors declare no other competing financial interests.

CRediT author contributions

Alexandra Jean: Conceptualisation, formal analysis, investigation, methodology, visualisation, writing (original draft, review and editing).
Jordan Funkhouser: Conceptualisation, investigation, project administration.
Andrew Makowski: Formal analysis, investigation, resources, writing (original draft, review and editing).
Stacy Groene: Resources, writing (review and editing).
Robert Brown: Conceptualisation, project administration, resources, supervision, writing (review and editing).

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