D. G. Hauser, A. Stokholm and T. H. Shellhammer

Investigating the potential for re-using "spent" dry-hops in the brew kettle

With the rising popularity of hop-forward American ales, dry-hopping has emerged as a ubiquitous technique for achieving pronounced hop aroma and flavor in beer. Due to the low temperatures at which dry-hopping is carried out (relative to kettle additions), hop-derived bitterness precursors are neither isomerized nor transferred to beer and remain, in significant quantities, within the discarded waste material. Therefore, this study was carried out to assess whether spent dry-hops could potentially be re-used for bittering. Spent hops following dry-hopping were collected from a local brewery, in addition to samples of the same pelletized hops used to dry-hop that specific brand. Hop acid utilization rates were measured on a lab scale using 1.5 L of un-hopped wort dosed separately with spent hops or hop pellets and boiled for 60 minutes in 5 L round-bottom flasks, and also on two pilot scale (~160 L) brews. Lab scale utilization rates for the hop pellets and spent hops were not significantly different, both averaging 29 %. On the pilot scale, utilization rates differed, at 21 % and 27 % for the hop pellets and spent hops, respectively. Finished pilot scale trial beers were then statically dry-hopped with Cascade hops at a rate of 386 g/hL (1 lb/bbl) for 48 hr at 13 °C (55 °F) in 50 L treatments, resulting in four beers for sensory analysis. The dry-hopped beers were found to be significantly different via an unspecified tetrad test, and the higher bitterness of the spent hop beer was confirmed via a two-alternative forced choice test. However, consumer testing for overall liking and liking of the beers' aroma and bitterness showed no significant differences between any pairwise comparisons of the four beers. These results demonstrate that from both an in-brewery utilization and organoleptic perspective, spent dry-hops could provide a feasible alternative to traditional kettle additions, while potentially saving brewers money and reducing environmental impact.

Descriptors: dry-hops, spent hops, sustainability, waste valorization, *humulus lupulus*, hop acid utilization

1 Introduction

The brewing industry is a large consumer of both water and energy, and generates significant quantities of solid and liquid waste. Compared to wine and spirits on a volume to volume basis, the carbon footprint of beer is substantially smaller, although on the basis of alcohol as a functional unit, spirits win out and wine performs nearly as well as beer [24]. One recent publication estimated in-brewery consumption of 8–12 kWH electricity, 5 hL water, and 150 MJ of fuel energy per hL of beer produced, in addition to the generation of ~13.6 kg of spent grains per hL of wort (35.3 lb/bbl), and 1.7–4.3 kg spent yeast per hL of beer (4.4–11 lb/bbl) [22]. These data point to a number of areas for potential improvement within brewing operations, although other life-cycle stages of beer production often have an even greater impact on sustainability.

According to the Oregon Consumption-Based Greenhouse Gas Emissions Inventory, the upstream emissions of beer consumption (including imports) in Oregon are equal to 202,700 metric tons of carbon dioxide equivalents annually, which is roughly equivalent to running 42,800 average passenger vehicles for a year [12]. A recent report commissioned by the State of Oregon Department of Environmental Quality reviewed 15 life-cycle assessment (LCA) studies dating back to 2005 in the US as well as various European and Asian countries [12]. On average, packaging (40 %) and raw material production (22 %, primarily barley) had the greatest relative carbon footprints. Retail and home refrigeration (18 %) was also a major contributor, with brewing operations (9 %), distribution (7 %), and waste management (4 %) making lesser contributions.

Brewers' spent grains (BSG) have received the most attention in terms of waste valorization within the brewing industry due to the large environmental impact of malted barley production, and their potential for re-use. BSG is available in large quantities year round; Brazil, the world's fourth largest producer of beer, generated 1.7 million tons of BSG in 2002, and a recent report estimated 76,000 tons of BSG are generated annually by Molson Coors alone [9, 21]. Given its high content of protein and cellulosic material, BSG may provide a valuable supplement for human nutrition, or a feedstock for the production of added-value bioproducts (vitamins, amino acids, etc.), polymers and resins [21]. Indeed, from the human nutrition perspective, BSG has already found its way into upcycled bread flour and health beverages.

The potential re-use of spent hops has garnered far less attention, likely due to the fact that most traditional beer styles call for

https://doi.org/10.23763/BrSc19-14hauser

Authors

Dean G. Hauser, Arnbjorn Stokholm, Thomas H. Shellhammer, Department of Food Science and Technology, Oregon State University, Corvallis, OR, United States; corresponding author: Tom.Shellhammer@oregonstate.edu

sparingly small quantities of hops compared to barley leading to less availability and smaller environmental impacts relative to barley [2, 17]. For instance, a study on the carbon footprint of New Belgium's Fat Tire Amber Ale found that only 0.2 % of the total carbon footprint came from hops [2]. However, this brand only uses 2.3 g of hops per six-pack of beer, which translates to roughly 1.08 g/L (0.3 lb/bbl). Hop-forward beer styles such as India Pale Ale (IPA), the production of which within the American craft beer market has increased nearly tenfold over the past decade, call for considerably more hop material, and it is not uncommon for American craft brewers to use up to or exceeding 20 g/L (~ 5 lb/bbl) for dry-hopping alone [17]. Dry-hopping, which involves the addition of hop material to partially or fully fermented beer, rather than to hot wort, is particularly inefficient in terms of the extraction of both bitterness precursors and aroma compounds from hop material [10, 16]. What's more, residual hop bitter acids and volatile aroma compounds found in spent dry-hops can likely be re-used in the brewing process, or in other industries. Essential oil from spent (likely kettle) hop material has been shown to be an effective repellent of insect food spoilers, while hop bitter acids have been investigated as potential antimicrobials for food preservation, and have potent miticidal effects on a major pest of honeybees [6, 14, 23].

One potential means for upcycling spent dry-hops in beer production is re-introducing them during kettle additions as a source of bittering hop acids. During kettle boiling, hop α-acids undergo a heat catalyzed isomerization to iso-α-acids, which are more bitter and soluble in beer, and are the primary drivers of beer bitterness [8]. The quantity of iso-α-acids in the hopped wort or finished beer relative to the quantity of α-acids added to the kettle is commonly referred to as hop utilization. This type of utilization is typically low (less than 40 %) for kettle additions and depends on a number of factors including pH, wort strength, kettle design, and notably, the form of the hop material [3, 4, 11, 20]. Given the likely compositional differences between spent dry-hops and pelletized hops, there may be differences in utilization rates, and/or the organoleptic qualities of beers alternately bittered with these materials. On the other hand, beers produced with "spent" hop upcycling could help brewers approach some of the sustainability challenges facing industry, while simultaneously allowing them to create a novel product that could differentiate them in a crowded market. As such, the goals of this project were to 1) investigate the bitterness utilization of spent dry-hops relative to pelletized hops on the lab scale 2) evaluate the efficacy of upcycling spent dry-hops to kettle additions from an organoleptic perspective in a pilot scale brewing system.

2 Materials and methods

2.1 Hops collection

Spent dry-hop slurry was collected from a local brewery along with samples of hop pellets that were used for the particular dry-hops addition. The hops consisted of five varieties (Citra®, Amarillo®, Centennial, Mosaic®, and Columbus) which were added in two additions over the course of five days of total hop-beer contact time, for a cumulative dosage of 1592 g/hL (4.125 lb/bbl). After this five day dry-hopping period, representative spent dry-hops samples were collected from production batches of a single brand of beer, brought to Oregon State University (OSU), dewatered using a mesh bag, and placed in high-barrier foil pouches which were subsequently purged with nitrogen, vacuum sealed, and stored at – 20 °C until analysis. This spent dry-hop slurry resulted from the production of an Imperial India Pale Ale (IIPA) which had a starting extract concentration of 17.3 °Plato, alcohol content of 8.4 % (v/v), real extract of 4.9 °Plato, color of 7.9 SRM, and pH of 4.76. Kettle hop additions were added at a rate of roughly 656 g/hL (1.7 lb/bbl) resulting in an ~ 80 Bitterness Unit (BU) beer. Whole cone Cascade hops used for pilot scale dry-hopping at OSU were obtained from Yakima Chief Hops (Yakima, WA).

2.2 Hop & beer analysis

2.2.1 Reagents and standards

HPLC-grade methanol was obtained from VWR International, BDH analytical (West Chester, PA, USA). Phosphoric acid was obtained from Avantor Performance Materials (Center Valley, PA). Ethyl ether and hydrochloric acid were obtained from EMD Millipore Corporation (Billerica, MA). International calibration extract ICE-3 and DCHA-Iso ICS-13 standards were obtained from ASBC. Carboxymethylcellulose and ethylenediaminetetraaceticacid were obtained from Sigma-aldrich (St. Louis, MO). Ammonium hydroxide and ammonium ferric citrate were obtained from Fisher Scientific (Fair Lawn, NJ).

2.2.2 HPLC analysis

The concentrations of hop acids and their derivatives in hops, wort, and beer samples were determined using ASBC methods Hops-14, Beer-23E, and Wort-23C under modified HPLC conditions on an Agilent 1200 HPLC [1]. Prior to analysis beer and wort was filtered through GHP Acrodisc® 13 mm syringe filters (Pall Corporation, East Hills, NY) in order to degas and remove solid particulate. Analysis was performed using a 2.6 µm EVO C-18 100 Å 100 x 4.6 mm LC 143 column (Phenomenex, Torrance, CA) held at 40 °C. 7 µL of each sample was injected and the elution was carried out using a flow rate of 1.6 mL/min. The solvent gradient was as follows: 10 % solvent A (reagent water): 90 % solvent C (75 % MeOH, 24.5 % H_2O, 0.5 % H_3PO_4) held for 5 min, then changed to 100 % solvent D (100 % MeOH) over 5 min and held for 2 min, then returned to 10 % solvent A: 90 % solvent C over 2 min, for a total run time of 14 min. α- and β-acids were measured at 314 nm. Iso-α-acids and humulinones were measured at 270 nm. All samples were injected in duplicate.

2.2.3 Beer and wort analysis

Physical properties of beer and wort such as density, extract, alcohol by volume (%ABV), and residual extract (RE) were measured using the Anton Paar DMA 4500 M (Anton Paar, Graz, Austria). Bitterness units were measured according to ASBC method Beer-23A [1]. Total polyphenols (TPP) were measured according to ASBC method Beer 35. Beer foam stability was measured according to the modified version of the European Brewery Convention (EBC) 9.42 method used by Kunimune and Shellhammer [15]. Foam measurements were carried out using a modified version of EBC method 9.42. A NIBEM-T meter was used, in conjunction with a Haffmans standard glass and Flasher Head. Prior to dispensing, glasses were washed with hot water and Alconox, then soaked in a solution of sodium tetraborate decahydrate (Borax) for 30 minutes, and thoroughly rinsed with deionized water. The beers, that had been attemperated to 14 °C, were dispensed directly from 20 L kegs pressurized with CO_2 to 2 bar through a Haffmans Inpack 2000 Flasher Head into the Haffmans standard glass (60 mm inside diameter, 120 mm inside height).

The foam-filled glass was placed in the NIBEM-T tester, and the NIBEM-T electrode allowed the foam to decay 10 mm, then the decay time to reach 10, 20, and 30 mm from this level was recorded as NIBEM-10, NIBEM-20, and NIBEM-30, respectively. Each beer was run on the NIBEM-T five times, and the NIBEM-30 value was collected. Data was collected on a single day, using storage-temperature beer (average ~14 °C) and at 1006 mbar atmospheric pressure and 42–46 % relative humidity.

2.3 Lab scale utilization trials

12 L of un-hopped wort was collected from a 2 hL brew at the start of kettle boil in 2 L glass bottles, which were stored at 4°C until use. The wort was produced using 96 % Rahr Premium Pilsner malt (Rahr Malting, Shakopee, MN) and 4 % Caramel Steam malt (Great Western Malting, Vancouver, WA), and had an extract concentration of 13.33 °P.

For each individual replicate, 1.5 L of wort was gravimetrically added to a 5 L round-bottom flask, in addition to either pelletized hops (PH) or spent dry-hops (SH), and outfitted with a distillation trap (Wilmad-LabGlass, Vineland, NJ) in order to minimize evaporation during wort boiling. Three replicates of PH and 4 of SH were carried out; the SH represented waste material from two separate production batches. Treatments were allowed to boil for 60 minutes, at which point distillate from the trap was added back to the round-bottom flask, and the round-bottom flask was cooled in an ice bath for 40 minutes to ~15 °C. Duplicate HPLC measurements on the PH, SH, and wort were made on the day of each trial.

2.4 Pilot scale beer production

330 L of un-hopped wort was prepared using a basic recipe for a pale ale made with 100 % Rahr Premium Pilsner malt (Rahr Malting, Shakopee, MN). The un-hopped wort was then split into two batches for kettle boil, which were alternately bittered with a single addition of SH or PH. Time from first hop strike to end of boil was 40 minutes in both cases. After boiling, hot break was separated using a whirlpool, and the wort was cooled, aerated, and fermented with ale yeast 1056 (Wyeast, Hood River, OR) at 20 °C. Extract concentrations prior to pitching yeast were 13.42 and 13.48 %w/w for the PH and SH worts, respectively. After fermentation, beers were filtered using a plate and frame filter with diatomaceous earth impregnated filter pads (HS2000, Pall Corporation, Port Washington, NY). Dissolved oxygen (DO) was monitored during filtration using an Orbisphere 3100 Portable Oxygen Analyzer (Hach, Loveland, CO) and ranged from 16–44 µg/L. Filtered beer was filled into standard kegs (15.5 gallons), and stored at 4 °C under 12 psi CO_2.

2.5 Pilot scale dry-hopping

A portion of each beer (PH and SH) from the pilot scale trial was dry-hopped. Dry-hopping was carried out based on the method established by Vollmer and Shellhammer [25]. 24 hours prior to dry-hopping, beer was removed from the cooler at 4 °C, and 50 L of beer was transferred into a modified 60 L stainless steel keg with a 4″ stainless steel opening fitted with a standard Sankey D-system coupler and modified spear (Sabco, Toledo, OH, U.S.A.). The beer was allowed to warm to approximately 15 °C prior to dry-hopping.

Whole cone hops were ground into a hop grist which was placed into a mesh bag (EcoBag, Ossining, NY), along with a stainless-steel fitting to ensure the hop charge sank to the bottom of the keg. The bags were stored inside high barrier pouches flushed with N2 until the dry-hopping event. For each dry-hop treatment, the keg was temporarily de-pressurized and opened under a stream of low-pressure CO_2. Simultaneously, the high barrier pouch was opened and the mesh bag containing ground hop grist was added to the beer. After the addition, the headspace was flushed with CO_2 and purged. After purging, the keg was inverted three times to ensure proper mixing. During dry-hopping, treatments were kept under 10psi CO_2 in a temperature controlled room held at 15 °C.

After 48 hours of dry-hopping, the beer was filtered in order to stop the dry-hopping process. Filtration was carried out using diatomaceous earth impregnated cellulose pads (HS2000, Pall Corporation, Port Washington, NY, U.S.A.). Dissolved oxygen (DO) was monitored during filtration using an Orbisphere 3100 Portable Oxygen Analyzer (Hach, Loveland, CO). Bright beer was not collected until DO was below 100 µg/L. After DO was within specification, bright, filtered beer was collected in a closed 19.6 L (1/6 US bbl) stainless steel keg with sufficient backpressure to reduce foaming. Between each filter run, filter pads were changed to prevent hops and beer carry-over from one treatment to the next. Filtered beer was stored at 4 °C and under CO_2 overpressure (11–12 psi) until chemical analysis.

2.6 Sensory analysis

2.6.1 Tetrad test

Dry-hopped beers produced from PH and SH (PH+ and SH+) were evaluated by sensory panelists in an unspecified tetrad test. Unspecified tetrad testing was chosen over other unspecified difference tests (triangle, duo-trio) due to recent work which has shown the tetrad to generally be more powerful, and require smaller sample sizes [7]. The panel consisted of 25 individuals, who ranged in age from 21–57, of which 20 were male and 5 were female. All panelists were self-identified beer drinkers. One hour prior to testing, beer was dispensed from an eight-head draft system (MicroMatic, Northridge, CA) at ~1.5 °C and 12 psi into pitchers. ~60 mL samples were then poured into 300 mL glasses and covered with plastic lids. Presentation order was randomized across panelists, each sample was assigned a random, three-digit blind code, and data was collected using paper ballots. Panelists were presented with four samples (two of each beer) and instructed to smell then taste samples, and group them into two groups of two based on similarity.

2.6.2 2-Alternative forced choice

Beers produced from PH and SH were evaluated in a 2-alternative forced choice (2-AFC) difference test. The panel was a subset of that in 2.6.1, and consisted of 20 individuals, covering the same age range, and including 15 males and 5 females. Randomization, data collection, and serving of samples was carried out as described in 2.6.1. During testing, panelists were instructed to taste each beer, and select the one that was more bitter.

2.6.3 Consumer sensory testing

All four beers (PH, SH, PH+, SH+) were subjected to consumer testing, which took place in three-hour blocks at two locations: a local bar (Location A), and the taproom of a local brewery (Location B). Combining both locations, 77 individuals aged 21–82 participated, of which 44 identified as male, 31 as female, and 2 preferred not to identify. All consumers identified as beer drinkers: 35 % reported daily consumption, 48 % weekly consumption, 13 % every other week, 3 % monthly, and 1 % every other month. Consumers were intercepted upon entering the testing sites, and asked if they would like to participate in a study involving smelling, tasting and rating their liking of four beer samples. All consumer data was collected on Asus Chromebook tablets using Qualtrics survey software (Provo, UT). Beers were assigned random 3-digit blind codes, were presented to panelists monadically, and presentation order was randomized across panelists by Qualtrics. At both locations, beer was dispensed cold via modified ice-filled coolers (“jockey boxes”) outfitted with ~15 meters (50 feet) of stainless steel piping (internal diameter 0.25 inches), beer shanks, and beer faucets. Panelists were presented with ~44 mL (1.5 oz) of each sample, which was served in 296 mL (10 oz) plastic cups or 148 mL (5 oz) glass cups at locations A and B, respectively. For each sample, panelists were asked to rate their aroma, overall, and bitterness liking on a 9-point hedonic scale, and to fill in open-ended question asking what they liked or disliked about each beer.

2.7 Statistical analysis

Mixed model analysis of variance (ANOVA) with post-hoc means comparisons (Tukey HSD), t-tests, and analysis of sensory difference test results were carried out using XLStat (Addinsoft Inc.). All difference test analysis was carried out using Thurstonian modeling, with binomial power functions.

3 Results and discussion

3.1 Lab scale utilization trials

Prior to pilot scale brewing, lab scale utilization trials comparing the relative bittering potential of spent dry-hops (SH) and pelletized hops (PH) were conducted in a model wort. The quantity of hop material added was calculated using previously collected data on PH and SH samples from the same brand of commercial beer, which indicated average, as is, α-acid content of 2 % and 10.5 % in the SH and PH, respectively, not accounting for moisture content. Dosage was scaled in both cases to result in ~50ppm iso-α-acids in the resulting wort assuming 30 % utilization. Data collected on the α-acid content of hop material on the day of the utilization trials indicated some deviation from these historical averages, with the PH α-acid content being slightly lower, and SH being slightly higher (Table 1). The resulting worts contained an average of 44 and 61 mg/L iso-α-acid in PH and SH, respectively. Utilization rates were then calculated on an overall basis using HPLC data on the wort before and after boiling and the added hop material, in addition to the density and mass of the cooled wort. Calculated utilization rates from both treatments ranged from 28–30 %, and were not found to be significantly different between PH and SH in a t-test ($p = 0.915$). Testing for practical equivalence of means using the “two-one-sided t-tests” (TOST) approach showed that utilization rates could be considered practically equivalent at a difference threshold of 2 % ($p = 0.014$). Although these results provide evidence that PH and SH have similar utilization rates, the authors caution against extending these results beyond the experiment in question. Brewers who may be interested in bittering with spent dry-hops would likely be best served by testing the technique within their specific brewing operations. Additionally, the collection, storage, and analysis of wet, spent hop material may require further investments in terms of labor, electricity, and instrumentation, respectively, which could limit the practicality of dry-hops re-use. Despite these caveats, the results do indicate that SH can potentially be used in place of PH for bittering additions from the standpoint that they can supply iso-α-acids to wort in a manner that is similar to PH.

Table 1 **Bitter acid composition of hop material and resulting wort in lab-scale (1.5 L) utilization trials**

	Hop pellets (PH)	Spent dry-hops (SH)
α-acid (%w/w)[a]	10.1 (2.4)	2.8 (3.2)
Hop material added (g)	2.2 (0.07)	11.4 (0.70)
Iso-α-acid (mg/L)[b]	43.7 (4.1)	61.3 (5.4)
Utilization (%w/w)[b]	28.7 (2.6)	28.8 (3.0)

Reported values are means, and percentages in parentheses are coefficients of variation (%cv)

[a] aggregate means of two sets of duplicate solvent extractions

[b] means of triplicate (PH) and quadruplicate (SH) utilization trials

Table 2 **Bitter acid composition of hop material and wort from pilot-scale (1.5 hL) utilization trial**

Hop material	Hop pellets (PH)	Spent dry-hops (SH)
α-acid (%w/w)[a]	12.1	3.1
Total kettle addition (g)	116	490
Dosed α-acid (mg/L)	91.1	98.5
Iso-α-acid (mg/L)	19.3	26.8
Utilization (%w/w)	21.2	27.2

[a] values are means of duplicate solvent extractions, standard deviation was less than 0.1 % w/w

3.2 Pilot scale beer production

Data from the lab scale trials indicated practical equivalence of utilization rates of PH and SH; however, this result was not obtained on the pilot scale (Table 2). It should be noted that the pilot scale brews were not replicated, and so no statistical conclusions can be drawn regarding differences in utilization rates or a lack thereof. Additionally, whereas lab scale trials were conducted in round-bottom flasks, pilot scale brewing took place on a steam-heated, 3hL system, which was equipped with an external calandria. As in the lab scale trials, bittering additions were scaled such that similar quantities of α-acids were added to the wort via either PH or SH, assuming equal utilization (Table 2).

The higher α-acid content in the SH and PH in pilot scale brewing relative to the lab scale trials stem from a slight change in the local brewery's recipe in terms of dry-hop additions between these two experiments, which was taken into account when planning the brews. Utilization rates were estimated using HPLC data on the added hop material and resulting worts, combined with brew-day data on the volume, extract concentration, density of wort before boiling, and the density and extract concentration of the cooled wort. Pilot scale brews resulted in 19.3 and 26.8 mg/L iso-α-acid in the PH and SH worts, and utilization rates of 21.2 and 27.2 % respectively. One possible explanation for the divergence of these utilization results from those on the lab scale could be inhomogeneity of the SH material. However, this seems unlikely given that the difference in α-acid content between duplicate samples of SH taken on three separate occasions (two for lab scale trials, one for pilot scale brewing) was consistently lower than 0.1 % w/w. Jaskula et al. observed better isomerization yields from a combination of non-isomerized hop extract and spent kettle hop material (0.30 % w/w α-acids) than the extract alone on a pilot scale, which supports our observation of better utilization of SH relative to PH [13]. Data previously collected on spent hops from the same brand indicate that an average of roughly 75 % of α-acids remained in the SH material relative to PH, accounting for moisture, and thus adding SH and PH at similar α-acid addition rates leads to considerably more vegetative material in the SH addition.

In terms of their chemical and physical properties, the two beers were nearly identical (Table 3), with measured original extract (%w/w), alcohol by volume (%v/v), and residual extract (%w/w) all falling within 0.1 % between the PH and SH beers. Foam stability, expressed as Nibem-30, or the time it takes for the foam to collapse 30mm from a set point, and total polyphenol content also showed only minute differences between the two beers. A slight decrease in iso-α-acid content was observed in both finished beers, which is a well-known consequence of fermentation [18], resulting in 18 and 23 mg/L iso-α-acids and 29 and 34 bitterness units in PH and SH beers, respectively. Compositional data on the pilot scale beers showed that aside from bitterness, the beers were nearly identical, suggesting that there were no negative consequences on fermentation performance with the use of SH, and that SH may be an effective substitute for PH in the brew-kettle.

Table 3 Compositional comparison of beers produced from hop pellets and spent dry-hops

Kettle addition	Hop pellets (PH)	Spent dry-hops (SH)
Original extract (%w/w)	13.42	13.48
Alcohol (%v/v)	5.87	5.90
Residual extract (%w/w)	4.80	4.80
Bitterness units	29	34
Iso-α-acid (mg/L)	18	23
Total polyphenols[a] (mg/L)	214	217
Nibem-30[b] (s)	291	287

[a] means of sample duplicates, standard deviation ranged from 4–7 mg/L

[b] means of quintuplicate measurements, standard deviation ranged from 11–17 seconds, temperature of measurements averaged 14.7 °C with 7.5 % coefficient of variation

Table 4 Unspecified tetrad and 2-AFC test results

Test	Unspecified tetrad[a]	2-Alterantive forced choice (2-AFC)[b]
Number of panelists	25	20
Guessing probability	1/3	1/2
Proportion of correct answers (%)	80	85
p value	< 0.0001	0.001

[a] test was run on dry-hopped beers PH+ and SH+

[b] test was run on PH and SH (not dry-hopped)

3.3 Sensory difference testing

Due to the measured differences in quantities of bittering compounds between the PH and SH beers that were mentioned in the previous section, beers were subjected to sensory difference testing prior to consumer sensory testing. Dry-hopped versions of PH and SH (PH+ and SH+) were evaluated for difference by the unspecified tetrad test. This test was carried out using the dry-hopped beers with the assumption that potential differences between PH and SH would be obfuscated to some extent by dry-hopping, and therefore that panelists would be less likely to detect a sensory difference between PH+ and SH+ than between PH and SH beers. Of the 25 individuals who participated, 80 % correctly grouped the samples, which provided significant evidence of a sensory difference ($p < 0.0001$) (Table 4). Many of the panelists also suggested that the SH+ beer was more bitter, which was in line with analytical measurements previously described.

Based on this feedback, a 2-alternative forced choice (2-AFC) test was carried out using the base PH and SH beers, in which panelists were asked which sample was more intensely bitter. Of the 20 panelists, 85 % identified the SH beer as more bitter, providing significant evidence of a difference between the two products ($p = 0.001$). These tests revealed that the PH and SH beers produced on a pilot scale were significantly different, and that the difference was related to the higher concentration of bitter compounds in the SH beer, which was confirmed analytically via BU and iso-α-acid testing. Although this result was not ideal from the perspective of proving the hypothesis that identical beers could be produced with kettle additions of SH in lieu of PH, it opened an interesting opportunity to subject these beers to consumer sensory testing.

3.4 Consumer sensory testing

Given that the SH performed in a similar manner to PH on both the lab and pilot scale in terms of utilization, and that the beers produced were significantly different from an organoleptic perspec-

Table 5 **Category phrases and corresponding ratings of the 9-point hedonic scale**

Scale value	Category phrase
9	Like extremely
8	Like very much
7	Like moderately
6	Like slightly
5	Neither like or dislike
4	Dislike slightly
3	Dislike moderately
2	Dislike very much
1	Dislike extremely

tive, all four beers (PH, SH, PH+, SH+) were subjected to consumer sensory testing. The goal of this testing was to see whether the use of spent hops for bittering would have an impact on consumer liking in terms of the aroma and bitterness quality of the beer, in addition to overall liking. Hedonic testing using a 9-point scale (Table 5) was chosen over several paired preference tests on dry-hopped and non-dry-hopped treatments given research that has shown that consumers often report preferences when testing two identical products [19]; although the products were found to be statistically different in this study, paired preference tests could prove misleading.

A wide range of consumer liking of the products was observed (Fig. 1) with nearly all of the attributes of all products receiving ratings ranging from 2 (dislike very much) to 9 (like extremely). Means across all attributes for all samples ranged from 5.61 to 6.18, and the median of every sample-attribute combination was 6 (like slightly). Analysis of variance (ANOVA) with a mixed model including panelist as a random effect and sample as a fixed effect was carried out for all three attributes (Table 6).

Significant panelist effects were found for all three liking attributes, and no significant sample effects were uncovered. This result indicates that while none of the four samples' liking ratings on any attributes were statistically different overall, liking ratings in general depended upon the panelist. This stands to reason, given the wide range of beer styles available to consumers, and the resulting wide range in particular preferences among consumers. While these data do not show differences between hedonic impressions of the samples, they do suggest that there were no striking defects or other organoleptic properties of the SH beers that would keep consumers from accepting a SH bittered beer.

4 Conclusions and implications

Spent dry-hops can be re-used in the brew kettle to supply hop bitterness in beer in a manner that is equally or more efficient than pelletized hops from a utilization perspective. Lab scale utilization trials in unhopped wort showed practical equivalence α-acid utilization between pelletized hops and spent dry-hop material collected from a local brewery. Pilot scale brewing (~ 160 L) yielded better utilization of spent dry-hops relative to pelletized hops, although brews were not replicated, so no statistical testing could be performed, and this difference may be attributed to process factors beyond the scope of the present study. From a sensory perspective, although the spent dry-hop bittered beers were significantly more bitter, no consumer preference was found in terms of aroma, overall, or bitterness liking between the two sets of beer. Taken together, these results indicate that spent dry-hops are a promising alternative to pelletized hops from both a brewing process and organoleptic perspective.

Given the near doubling in American craft brewers' hopping rates over the past decade, and the rise to prominence of hop-forward beer styles which require considerable quantities of dry-hops [17], brewers are increasingly needing to investigate potential methods for dry-hops waste management. Additionally, the resource-intensive nature of hop produc-

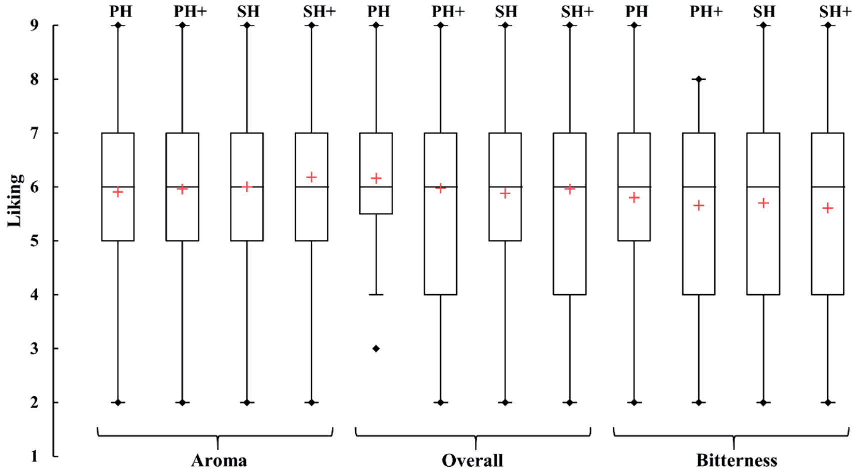

Fig. 1 **Boxplots of consumer aroma, overall, and bitterness liking of beers bittered with pelletized hops (PH), spent dry-hops (SH) and dry-hopped PH and SH beers (PH+, SH+)**

Table 6 **Analysis of variance table for consumer data**

			Aroma liking		Overall liking		Bitterness liking	
Source	**Type**	**DF**	**F-statistic**	**p-value**	**F-statistic**	**p-value**	**F-statistic**	**p-value**
Panelist	Random	76	2.7	< 0.0001	2.0	< 0.0001	1.6	0.006
Sample	Fixed	3	0.8	0.50	0.4	0.73	0.3	0.86
Error		223						

tion in terms of water and energy in irrigation and the use of fossil fuels for preservation of hop material via kiln-drying, respectively, indicate potential environmental benefits associated with more efficient use of hop material. Further, beer consumers have been reported to be willing to pay more for sustainably brewed beer [5], and the replacement of pelletized hops with spent dry-hops may provide brewers with considerable cost savings. Of course, the potential benefits of dry-hops upcycling may be precluded by energy consumption if the hop material needs to be stored under refrigeration for long periods of time before use, and may not be possible due to complex logistics and planning involved in large scale breweries. Therefore, further research may be needed on storage stability of spent dry-hops or possible methods of preservation. Nonetheless, the re-use of spent dry-hops for bittering is a promising new technique, and may provide an opportunity, particularly for smaller-scale brewers, to create novel new products while cutting costs, and potentially lowering their environmental impact.

Acknowledgements

The authors would like to thank Karli Van Simaeys and Keisha Harrison for their assistance in conducting the sensory trials, and Jeff Clawson for his guidance and assistance in brewing the pilot scale beers. Additionally, the authors would like to express their gratitude to Nick Arzner, Matt Williams, and Block 15 Brewing Co. for their support.

5 References

1. American Society of Brewing Chemists: Hops – 14, α-acids and β-acids in hops and hop extracts by HPLC (International Method); Beer-23A, Bitterness Units (International Method); -23E iso-α-acids in beer by HPLC; -35 Total Polyphenols (International Method); Wort-23C Iso-α-acids in Wort by HPLC, in Methods of Analysis, 8 ed., American Society of Brewing Chemists, St. Paul, MN, (1992).
2. The Carbon Footprint of Fat Tire Amber Ale,The Climate Conservancy, 2008.
3. B. Jaskula-Goiris; L. De Cooman and Goiris, K.: *Humulus Lupulus*: Hop Alpha-acids Isomerization – A Review, BrewingScience, **71** (2018), no. 11/12, pp. 85-95.
4. Briggs, D. E.; Boulton, C. A.; Brookes, P. A. and Stevens, R.: 8 – The chemistry of hop constituents in Brewing, Woodhead Publishing, 2004.
5. Carley, S. and Yahng, L.: Willingness-to-pay for sustainable beer, PLOS ONE, **13** (2018), no. 10, pp. e0204917.
6. Degrandi-Hoffman, G.; Ahumada, F.; Probasco, G. and Schantz, L.: The effects of beta acids from hops (*Humulus lupulus*) on mortality of Varroa destructor (Acari: Varroidae), Experimental & Applied Acarology **58** (2012), no. 4, pp. 407-421.
7. Ennis, J. M. and Jesionka, V.: The Power of Sensory Discrimination Methods Revisited , Journal of Sensory Studies, **26** (2011), no. 5, pp. 371-382.
8. EBC Technology and Engineering Forum, Hops and Hop Products: (manual of good practice), Getränke-Fachverlag Hans Carl, 1997.
9. Garcia-Garcia, G.; Stone, J. and Rahimifard, S.: Opportunities for waste valorisation in the food industry – A case study with four UK food manufacturers, Journal of Cleaner Production, **211** (2019), pp. 1339-1356.
10. Hauser, D.G.; Lafontaine, S.R. and Shellhammer, T.H.: The extraction efficiency of hop bitter acids and volatiles during dry-hopping, Brewing Summit, 2018, San Diego, CA,
11. Held, R.: Hop Products: Extracts, Pellets, and Modified Alpha, Beta Acids, MBAA TQ, **35** (1998), no. 3, pp. 133-140.
12. Heller, M.: „Beer",Food Product Environmental Footprint Literature Summary, Center for Sustainable Systems, University of Michigan, 2017.
13. Jaskula, B.; Goiris, K.; Van Opstaele, F.; De Rouck, G.; Aerts, G. and De Cooman, L.: Hopping Technology in Relation to α-Acids Isomerization Yield, Final Utilization, and Stability of Beer Bitterness, Journal of the American Society of Brewing Chemists, **67** (2009), no. 1, pp. 44-57.
14. Kramer, B.; Thielmann, J.; Hickisch, A.; Muranyi, P.; Wunderlich, J. and Hauser, C.: Antimicrobial activity of hop extracts against foodborne pathogens for meat applications, Journal of Applied Microbiology, **118** (2015), no. 3, pp. 648-657.
15. Kunimune, T. and Shellhammer, T. H.: Foam-Stabilizing Effects and Cling Formation Patterns of Iso-α-acids and Reduced Iso-α-acids in Lager Beer, Journal of Agricultural and Food Chemistry, **56** (2008), no. 18, pp. 8629-8634.
16. Lafontaine, S. R. and Shellhammer, T. H.: Impact of static dry-hopping rate on the sensory and analytical profiles of beer, Journal of the Institute of Brewing, **76** (2018), no. 3, pp. 199-208.
17. Lafontaine, S. R. and Shellhammer, T. H.: How Hoppy Beer Production Has Redefined Hop Quality and a Discussion of Agricultural and Processing Strategies to Promote It, MBAA TQ, **56** (2019), no. 1, pp. 1-12.
18. Laws, D. R. J.; McGuinness, J. D. and Rennie, H.: The Losses Of Bitter Substances During Fermentation, Journal of the Institute of Brewing, **78** (1972), no. 4, pp. 314-321.
19. Marchisano, C.; Lim, J.; Cho, H. S.; Suh, D. S.; Jeon, S. Y.; Kim, K. O.and O'Mahony, M.: Consumers Report Preferences When They Should Not: A Cross-Cultural Study, Journal of Sensory Studies, **18** (2003), no. 6, pp. 487-516.
20. McMurrough, I.; Cleary, K. and Murray, F.: Applications of high-performance liquid chromatography in the control of beer bitterness, Journal of the American Society of Brewing Chemists, **44** (1986), no. 2, pp. 101-108.
21. Mussatto, S. I.; Dragone, G. and Roberto, I. C.: Brewers' spent grain: generation, characteristics and potential applications, Journal of Cereal Science, **43** (2006), no. 1, pp. 1-14.
22. Olajire, A. A.: The brewing industry and environmental challenges, Journal of Cleaner Production, (2012).
23. Rój, E.; Tadić Vanja, M.; Mišić, D.; Žižović, I.; Arsić, I.; Dobrzyńska-Inger, A. and Kostrzewa, D.: Supercritical carbon dioxide hops extracts with antimicrobial properties, Open Chemistry, **13** (2015), no. 1.
24. Saxe, H.: „LCA-based comparison of the climate footprint of beer vs. wine & spirits. Fødevareøkonomisk Institut, Københavns Universitet",Report, No. 207 (2010).
25. Vollmer, D. M. and Shellhammer, T. H.: Influence of Hop Oil Content and Composition on Hop Aroma Intensity in Dry-Hopped Beer, Journal of the American Society of Brewing Chemists, **74** (2016), no. 4, pp. 242-249.

Received 7 May 2019, accepted 12 June 2019