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Comparative analysis of dry-hopping with hop extracts versus hop pellets

This study investigated the use of hop extracts as a viable alternative to traditional T90 hop pellets in the brewing process of American-style pale ales. Dry hopping with hop extracts presents a potential solution to challenges associated with beer losses due to hop matter absorption, particularly as craft brewers increasingly employ larger hop bills. The objective was to assess the feasibility of hop extracts in maintaining the physical and sensorial integrity of the final product while potentially reducing beer losses and increasing overall brewing efficiency. This comprehensive analysis revealed that beers brewed with hop extracts exhibited physical attributes, such as bittering, beer volatile composition, and accelerated ageing behaviour, similar to those utilizing T90 pellets. Sensorial data exhibited strong similarities, with dominant stone fruit aromas and a complex flavour profile which included citrus, fruity, floral, spicy and grassy characteristics. Furthermore, the usage of fully soluble hop extracts in lieu of hop pellets increased beer yield by $10.19 \pm 2.25\%$, by removing the requirement for dry-hop matter removal. The similarities observed between the two dry hop additions demonstrated the potential utilization of hop extracts as a substitute or complementary ingredient for brewers crafting hop-heavy beers. In conclusion, this study demonstrated the application of hop extracts in creating more efficient, hop-forward beers. Hop additions with hop extracts have the potential to create high quality beers, stability under aged conditions, and continuity of brews resulting in process optimization, increased efficiency and enhanced profitability.

Descriptors: hops, hop extracts, efficiency, stability, dry hop

1 Introduction

Among the variety of components contributing to beer's sensory profile, hops are an essential contributor to both aroma and flavour. The technique of dry hopping, a post-fermentation addition of hops, allows brewers to infuse their brews with significant aroma and flavour thereby enhancing the complexity and appeal of beers such as pale ales and India Pale Ales (IPAs) [1, 2].

Dry hopping, while offering exceptional aroma and flavour benefits, presents challenges in terms of beer losses due to the absorption of liquid by hop matter. In recent years, as craft brewers have increasingly employed larger hop bills to achieve desired sensory attributes, this issue becomes more pronounced, leading to increased waste and reduced profitability [3, 4]. The evolution of brewing practices has provided a solution to this problem: hop extracts. Extracts offer the potential to mitigate beer losses by virtue of their liquid form, eliminating the need for the removal of wet hop waste. Furthermore, their fully soluble, concentrated nature means

that smaller volumes can potentially achieve the same sensory impact, streamlining the brewing process [5, 6, 7, 8, 9].

Hop extracts have been available for many years and although the concept of utilizing hop extracts presents itself as a promising solution to the issues posed by traditional hop pellets, comprehensive research comparing the effects of brewing with hop extracts against hop pellets is notably scarce. The present study aimed to bridge this knowledge gap by investigating the impact of hop extracts and T90 hop pellets on the brewing process and final beer quality. Using physical and sensory analysis techniques, direct comparisons of American-style pale ales brewed with both hop pellets and hop extracts was achieved.

The primary objective of this study was to compare the viability of brewing with hop extracts in comparison to traditional T90 hop pellets. Specifically, the study aimed to assess the feasibility of hop extracts as a substitution for hop pellets in terms of maintaining the physical integrity of the final product while potentially mitigating beer losses. This study assessed beer loss reduction, haze contribution, bittering content, extent of fermentation and alcohol content, as well as the hop volatiles present in the final product and overall sensorial experience.

By systematically investigating these parameters, this study provided valuable insights into the potential advantages and disadvantages associated with utilizing hop extracts as a substitute for hop pellets in brewing practices. This research endeavoured to advance brewing knowledge and help brewers make informed decisions for optimizing the brewing process, using hop extracts

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to achieve the same desired attributes as using T90 pellets while increasing the efficiency, profitability and reducing waste associated with traditional brewing processes.

2 Materials and methods

2.1 Hop Pellets and Extracts

Nectaron® hop pellets, 2022 (22186-1) batches, were sourced from New Zealand Hops Ltd (T90 pellets, New Zealand). The pellets were processed to produce a solution of steam distilled hop oils suitable for dry hop addition in the brewing process detailed below.

2.2 Chemicals

The analytical reagents were sourced from Fisher Scientific™, isopropanol (propan-2-ol), > 99.8 % purity (HPLC grade, UK); Honeywell Riedel-de-Haën™, isooctane (2,2,4-trimethylpentane), > 99 % purity (Germany); Atom Scientific Ltd., hydrochloric acid, 35.0 – 36.6 % purity, food grade (UK); Sigma Aldrich™, sodium chloride, > 99 % purity (Switzerland); Air Products Plc, helium CP grade, 99.999 % (N5.0) purity (UK).

2.3 Brewing methodology

Two American-style pale ales were brewed using the following method. Initially, a 23 L batch of wort was produced with a grain bill of Crisp Malt Lager (3900 g) and Crisp Dextrin Malt (430 g). Mash was performed at 67 °C for 60 minutes, followed by 10 minutes at 75 °C. Chinook T90 pellets (4.0 g), were added for bittering during the boil, for 60 minutes at 100 °C. Subsequently, Citra (36.0 g), Chinook (36.0 g), Cascade (36.0 g) T90 pellets were added during the whirlpool stage to provide aroma. After cooling, the brew was fermented using Safale® US-05 (11.5 g) at 19 °C for 9 days. The beer was then split into two 8 L batches for dry hopping. The first batch, the T90 Pellet Brew (TPB), was dry hopped for 48 hours using Nectaron® T90 pellets (37.5 g), followed by coarse filtration. Beer losses (w/w) were recorded. The second batch, the Hop Extract Brew (HEB), was dry hopped using Nectaron® hop extracts (12.5 g); hop oil addition was standardized using oil extraction yield data and published utilization rates [10]. Due to the soluble nature of hop extracts no filtration step was required. Both brews were then carbonated in 18 L Cornelius kegs, using carbon dioxide at 11 – 12 PSI (estimated 4.9 g/L dissolved carbon dioxide). Samples were then bottled in 330 mL brown swing top bottles. The brews were conditioned at 4.0 °C for two weeks prior to analysis. The brewing procedure was performed in duplicate and subsequent results were calculated as an average.

2.4 Physico-Chemical Analysis

2.4.1 Analysis of beer volatiles

The Gas Chromatography-Mass Spectrometry (GCMS) analysis was conducted utilizing an Agilent 5973 Gas Chromatograph – Mass Spectrometer Detector, coupled with HTA HT2800T Autosampler, consisting of GC oven 6890, inert MSD 5973 and 7683), BP5MS 5 % Phenyl Polysilphenylene Siloxane 30 m (l)

x 0.25 µm (i.d) GC column, 10 µL injection syringe (Agilent p/n 9301-0713), 4 mm i.d tapered focus liner (Agilent p/n 210-4022-5). Solid Phase Microextraction (SPME) analysis was performed on a 5.0 mL sample of beer, saturated with 2.0 g of NaCl. The sample was heated to 40 °C and agitated for a duration of 30 minutes. The 10 cm PDMS/DVB/CAR fibre was injected into the sample vial headspace and exposed for a duration of 30 minutes. The fibre was then injected directly into the GCMS for compound identification. The resulting spectrum was integrated by Agilent ChemStation software (version E.02.02.1431). A supplied NIST spectral library was used to assist with the identification of components. Analyses were performed in triplicate and used to calculate averages and standard deviations.

2.4.2 Bittering analysis

Beer bitterness, expressed in International Bitterness Units (IBUs), was quantified using EBC methodology 9.8 Bitterness Units (BU) of beer. The analysis was conducted using a UV1720 Single Beam UV-VIS Spectrophotometer. Extraction of bittering compounds was performed by combining 10 mL of degassed beer with of 20 mL of acidified iso-octane which was then agitated at 130 rpm for a duration of 15 minutes, at 20 °C. A sample of iso-octane was pipetted into a cuvette for analysis at 275 nm for quantification of bittering content. All analysis was performed in triplicate.

2.4.3 Alcohol content

The alcohol by volume (ABV %) was calculated using specific gravity measurements, utilizing a Mettler Toledo DA-100M Density Meter. Sample volumes of 5.0 mL were analysed in triplicate at 20 °C. The ABV (%) content was then calculated using the following equation, where OG denotes Original Gravity and FG denotes Final Gravity:

$$ABV (\%) = (OG - FG) \times 131.35$$

2.4.4 Haze

The turbidity of the beers was measured using the Hach turbidity meter 2100QISO. Vials of 0, 100, and 800 NTU StablCal® sourced from Hach were used as calibration standards. Turbidity analysis, expressed in FNU (Formazin Nephelometric Units), was performed on 10 mL samples, and results recorded in triplicate. Conversion from FNU units to EBC haze turbidity units was calculated using the following formula:

$$1 \text{ EBC} = 4 \text{ FNU}$$

2.5. Sensory Assessment

Descriptive sensory evaluation was performed in triplicate to characterize the aroma and flavour of TPB and HEB samples. A total of four experienced tasting panellists (1 female and 3 males) participated. Flavour and aroma quantitative descriptive analysis was performed in triplicate; a scale of 0 – 9 was used to measure specific attributes; fruity – tropical, fruity – stone fruit, fruity – berries, citrus – lemon, citrus – other, floral, herbal, spicy, grassy, malty, woody, resinous and sulfur (e.g. off-notes, DMS, etc.). Additional

sensory attributes of bitterness, sourness, body and linger were also assessed.

2.6 Accelerated Ageing

The beer shelf life was assessed using a methodology described by Lehnhardt et al, where the beer underwent forced ageing by exposure to elevated temperatures [11]. The bottled samples were agitated for 24 hours and then submerged in a Thermoscientific Circulating Water Bath U01367, at 40 °C for 4 days, equivalent to 3–5 months of natural ageing. The samples were then reassessed on all the previously described criteria: beer volatiles, bittering, alcohol, haze content and sensory assessment.

3 Results and discussion

3.1 Beer Losses

Within the context of the brewing process, the application of dry hopping with T90 hop pellets required a subsequent filtration step to eliminate residual hop particulates that remained undissolved within the beer. In this investigation, a coarse filtration approach was employed on an 8.00 KG batch of TPB to remove these particles. This resulted in beer losses of 0.81 ± 0.18 KG. In contrast, the utilization of hop extracts, characterized by their liquid nature and full solubility, eliminated the need for filtration in the case of HEB. As a direct consequence, the utilization of hop extracts resulted in 10.19 ± 2.25 % more beer being recovered when compared to dry hopping with T90 pellets. In a commercial setting, the resultant increase in beer production per brewed batch may provide a potential route to enhanced brewery profitability. Additionally, substitution with soluble hop extracts would reduce the requirement for filtration and/or centrifugation equipment to remove insoluble hop matter, while simultaneously facilitating the full dispersion of aromatic compounds. Other innovative techniques that aim to reduce beer losses in hop-heavy beers include utilizing a rotary mixer with a jet to accelerate mixing and therefore dispersion of hop components into beers, increased contact time of hops in static dry-hopping and purging of fermentation or maturation tank with carbon dioxide. However, these methods have the disadvantages of high capex investments as well as potential increases in herbaceous and astringency characteristics due to increased extraction of polyphenols and other hop derived compounds [12, 13, 14, 15]. Dry-hopping with hop extracts provides a low-cost route to reduce dry-hop related beer losses whilst ensuring full dispersion of pure essential hop oil components.

3.2 Solid-Phase micro-extraction and beer volatiles

The utilization of Gas Chromatography-Mass Spectrometry Solid Phase Microextraction (GCMS-SPME) allowed for the identification of volatile compounds present in the beers, enabling an understanding of compositional changes over the duration of the brewing process [16, 17, 18].

As seen in figure 1, the initial analysis of the wort, prior to any aroma hop addition, revealed the presence of five major volatile components: isoamyl acetate, myrcene, ethyl hexanoate, phenethyl alcohol, and ethyl octanoate. Upon introduction of dry-hop additions, an incorporation of hop-derived compounds was observed in both the HEB and TPB. Specifically, the compounds linalool, β -caryophyllene, and α -humulene were consistently identified. The application of area-under-peak measurements from the GCMS-SPME chromatograms provided a perspective on the volatile content within the beers. HEB exhibited higher overall volatile levels, possibly indicating more intense aromas, attributed to the elevated contributions of myrcene, ethyl octanoate, β -caryophyllene, and α -humulene. After being exposed to accelerated ageing conditions both HEB and TPB exhibited an overall reduction in the volatile content, according to the area-under-peak measurements, suggesting a potential decrease of hop-related flavours. Notably, myrcene, a key hop-derived compound, exhibited the most significant decrease, suggesting it was more sensitive to the ageing process and which was possibly related to its higher volatility relative to other hop-derived compounds [19, 20].

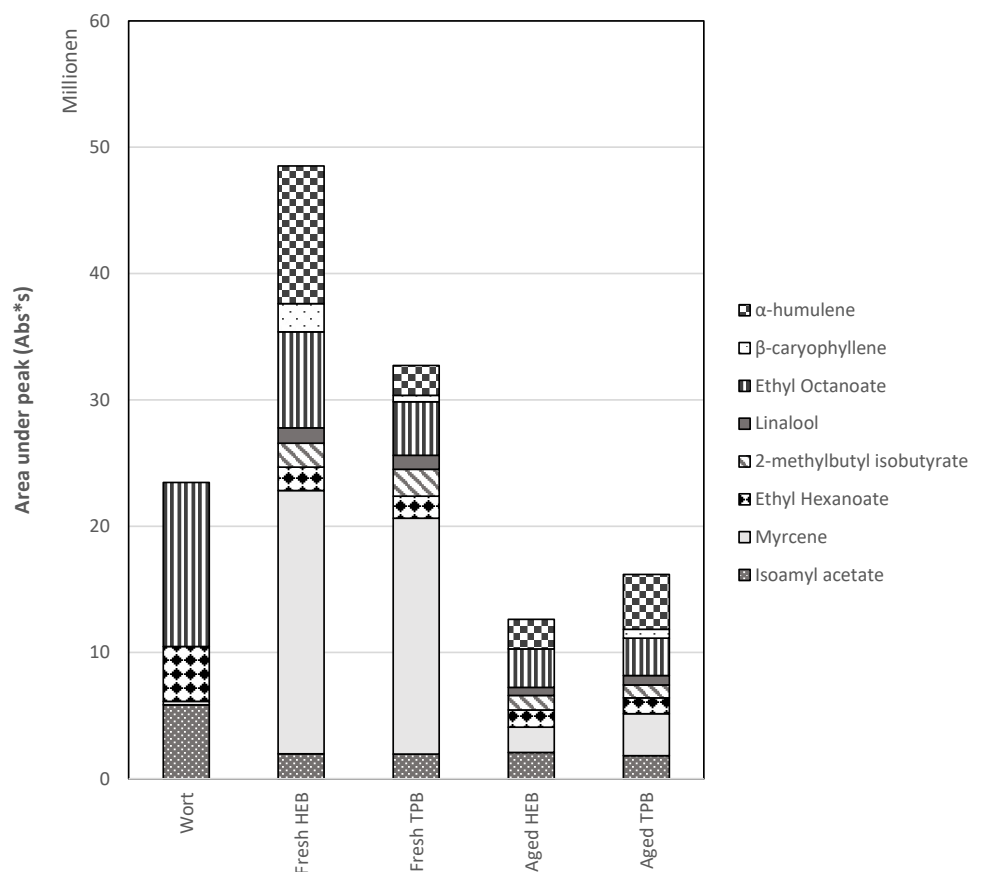


Fig. 1 GCMS-SPME analysis of beer volatile components of wort, as well as HEB and TPB samples, before and after accelerated ageing

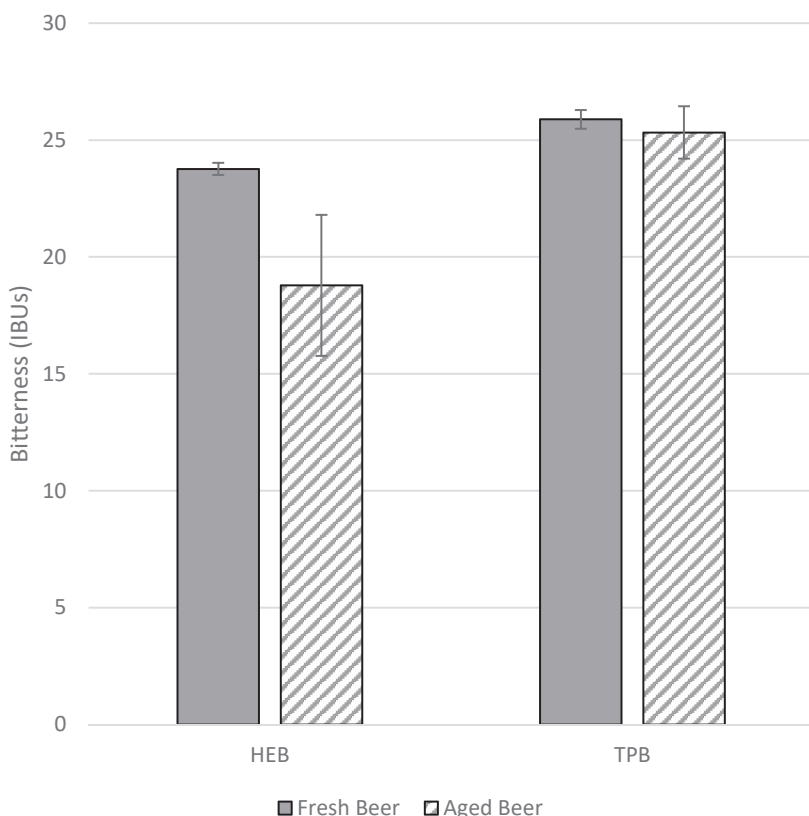


Fig. 2 Measured bitterness in HEB and TPB samples, before and after accelerated ageing

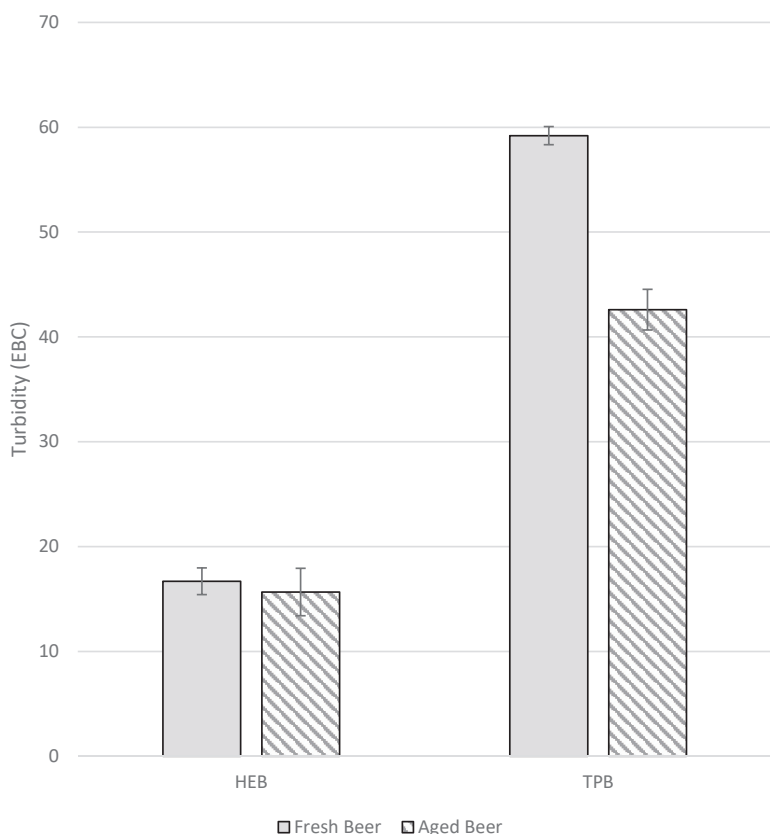


Fig. 3 Turbidity in EBC units of HEB and TPB samples before and after accelerated ageing

The investigation into volatile compounds through GCMS-SPME offered valuable insights into the evolution of aromatic profiles of TPB and HEB over the duration of brewing and following forced ageing conditions. The identification of volatile components provided by dry-hop additions and accelerated ageing provided enhanced understanding volatile composition of hop-derived compounds.

3.3 Bitterness

During the boil stage of the brew, hop-derived alpha acids are isomerized into iso-alpha acids, contributing towards bitterness in the final beer. High temperature is a key trigger for this reaction; therefore, the dry-hop additions were not expected to impart significant amounts of bitterness to the beer [21]. Quantification of bittering content showed that HEB contained a lower measured bitterness than TPB, by 3.7 IBU. Due to the lack of additional isomerized acids, the additional bittering may be provided by other compounds that also absorb at 275 nm. This additional absorbance is unlikely to be provided by essential oil components, which are found in the dry-hop extract, and therefore may be due to the polyphenol, humulinone and hulupone content found in hop pellets [22, 23, 24, 25, 26].

Following forced ageing of brews, the measured bitterness of both beers decreased – HEB by a greater extent than TPB. This additional decrease was likely due to oxidation and further reactions of the iso-alpha acids [27, 28].

3.4 Specific gravity and ABV (%)

The assessment of fermentation and resultant alcohol content involved the utilization of specific gravity values measured pre-fermentation (OG), post-fermentation (FG), and following the accelerated ageing process (ASG). These measurements, as shown in table 1, were used to calculate the ABV %, yielding a determined value of 4.3 ± 0.0 % for both the HEB and TPB. These final ABV % values slightly deviated from the range prescribed by the Brewer’s Association Style Guidelines, which stipulate a range of 4.4 – 5.4 % for American-style pale ale. Additionally, it was observed that the OG of the wort fell below the acceptable range outlined in the Brewers Association guidelines for 2023 (1.044 – 1.050) [29]. This suggested that the lower-than-expected ABV % values may have been associated with non-dry hop related, pre-fermentation parameters, such as diastatic power, malt batch variability, suboptimal mash extraction, or temperature fluctuations within the brewing system [30, 31, 32, 33].

Upon undergoing accelerated ageing, the specific gravity profiles were consistent for both HEB and TPB. This behaviour indicated the absence of any

Table 1 Specific gravity measurements of HEB and TPB pre- and post-fermentation, and after accelerated ageing

Specific Gravity	HEB	TPB
Original Gravity (OG)	1.040 ± 0.001	1.040 ± 0.001
Final Gravity (FG)	1.005 ± 0.000	1.005 ± 0.000
Post-ageing Specific (ASG)	1.005 ± 0.000	1.005 ± 0.000

ongoing yeast activity within the samples. The ABV % remained constant for both brewing methodologies, indicating that the impact of hop extract utilization in brewing did not differ from that of pellets.

The results demonstrated that dry hopping with hop extracts mirrored the behaviour of T90 hop pellets, with neither approach influencing the extent of fermentation or the final alcohol content of the beverage. It also remained consistent for the aged beers. These findings demonstrated the consistency of the fermentation process and ABV % regardless of the form of the dry-hop addition.

3.5 Turbidity and Haze

The significance of haze in beers has evolved over time. While historically haze was regarded as a defect in low-quality beers, contemporary perspectives recognize haze as a sought-after attribute in certain modern pale ales. Haze in beer can arise from diverse factors, including the interaction of sizable haze-active molecules sourced from malt and hops, such as proteins and polyphenols, polymerization of polyphenols and or contributions from residual yeast content due to the absence of pasteurization or sterile filtration [34, 35, 36, 37].

Turbidity measurements of the HEB and TPB were recorded, following the brewing and accelerated ageing processes (Fig. 3). The resultant beers displayed notably different haze values, with TPB exhibiting an initial turbidity approximately four times greater than HEB. This variation may be attributed to differences in the hop additions' composition, as the hop extract exclusively contained hop-derived volatile oils, thereby lacking the hop-derived polyphenols conducive to haze formation [38, 39, 40].

Upon undergoing accelerated ageing, HEB exhibited a 6.15 % reduction in turbidity, while TPB's turbidity decreased by 28.0 % with higher batch-to-batch variability. The phenomenon of decreased beer turbidity following storage and accelerated ageing has been documented previously [41, 42, 43, 44].

The application of forced ageing serves as an informative tool in assessing beverage stability, particularly in evaluating potential challenges related to colloidal instability and the quality of haze content during prolonged storage. The data emphasized that over time, the HEB haze was more stable compared with TPB, and would allow brewers to create a more stable beer throughout shelf life [36, 45, 46].

3.6 Sensory Assessment

Flavour and aroma sensory profiles were quantitatively assessed for HEB and TPB, as well as after the samples had undergone accelerated ageing (Fig. 4 and 5). HEB and TPB both exhibited strong overall fruity characteristics, with stone fruit being a dominant characteristic, as well as notable tropical and berry notes. In terms of aroma, TPB and HEB have similar fruity characteristics and intensities. This is reflective of the similarities in ester composition.

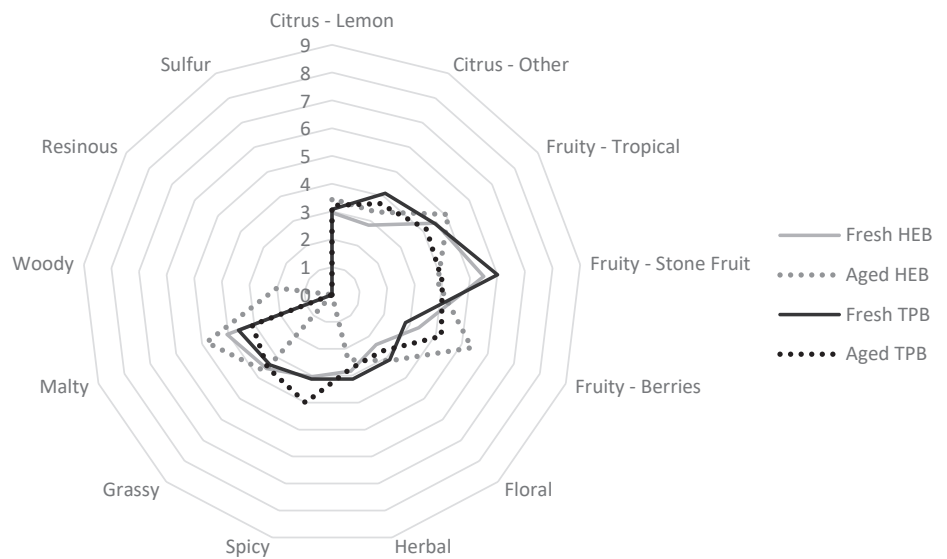


Fig. 4 Quantitative descriptive aroma profiling of HEB and TPB samples, before and after accelerated ageing

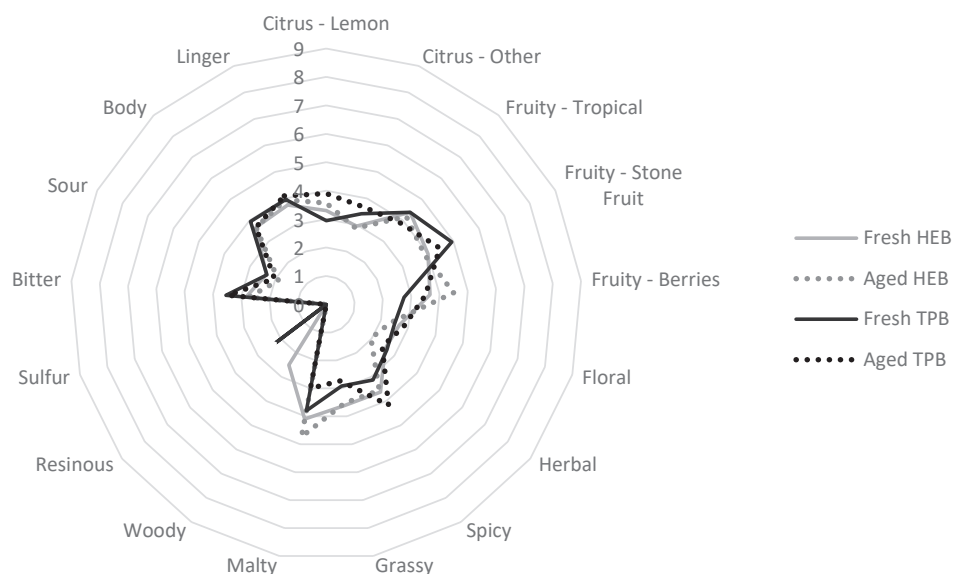


Fig. 5 Quantitative descriptive flavour profiling of HEB and TPB samples, before and after accelerated ageing

tions, compounds that frequently provide fruity aromas, shown the volatile analysis in Section 3.2. Solid-Phase micro-extraction and beer volatiles [47, 48]. Sensory analysis indicated strong similarities in tropical flavour, however T90 pellet addition resulted in higher stone fruit intensities whereas hop extract addition provided more intense berry flavours. Recent research has shown that hop-derived thiols may also contribute to the fruity flavours and aromas found in beers, such as black-currant-like 4-methyl-4-sulfanylpentan-2-one (4MSP) and passionfruit/grapefruit-like 3-sulfanylhexan-1-ol (3SH), which may be a factor contributing to perceived flavour differences observed. However, thiols have been shown to be present at very low concentrations, some as little as nanogram per litre, which may be below the limit of detection of the GCMS-SPME method outlined above [49, 50, 51, 52, 53].

The overall sensory profiles between TPB and HEB exhibited comparable trends; further sensorial similarities, with differences of < 1 in perceived intensities of specified flavour and aroma attributes, included citrus – lemon, floral, herbal, spicy, grassy, malty, woody and resinous. Additionally, both brews completely lacked sulfur-related off-notes, and showed consistent similarities in perceived bitterness, sourness, body and linger intensities.

Interestingly, the perceived bitterness was slightly less intense for HEB than TPB, which confirms the physical data obtained in Section 3.3 Bitterness, indicating that small amounts of polyphenols, humulinones and hulupones may have been contributing to this attribute in TPB [22, 23, 24, 25, 26]. Two further differences in the flavour of the beers were resinous, which was identified in TPB, and woody, in HEB. The latter may be attributed to the differences in α -humulene composition in the beers, which was identified in Section 3.2. Solid-Phase micro-extraction and beer volatiles, a compound known to contribute woody notes [54].

Following accelerated ageing the sensory profiles of both beers predominantly maintained similar sensory profiles, with differences in intensity < 1; citrus – lemon, citrus - other, fruity – tropical, floral, herbal, grassy, malty, resinous, sulfur-related off-notes, bitterness, sourness, body and linger exhibited only small changes. Both TPB and HEB fruity aromas behaved similarly under ageing conditions; stone fruit attributes decreased while the berry notes increased. However, differences were seen between the two brews; HEB saw a decrease in spicy aromas and a simultaneous increase in woody yet decrease in woody aromas whereas TPB showed an increase in spicy flavours and a decrease in resinous flavours. Described sensorial differences may be related to the oxidation and volatilization of hop related compounds, which has been observed in previous beer ageing research [55, 56, 57, 58, 59].

Overall brewing with hop extracts or T90 pellets provided predominantly similar sensory experience, which largely behaved similarly when exposed to forced ageing conditions. The data shows that by weight three times more pellets and needed, when compared to hop extracts, to deliver a similar sensorial experience. Some differences in sensorial impact were noted in relation to bitterness, fruitiness, woody and resinous attributes in finished beer, and further differences in woody, spicy and resinous characteristics in aged beer. This research provided informative data to support brewers in the application of pellet substitution with hop extracts,

to allow for sensory optimization for final beverage product and during storage.

4 Conclusion/Summary

This study provided a comparative analysis of the physical and sensory aspects of pale ales brewed using dry-hopped Nectaron[®] hop extracts and traditional T90 Nectaron[®] pellets. The similarities observed between the two dry-hop additions demonstrated the potential utilization of hop extracts as a substitute or complementary ingredient, offering a viable alternative for brewers seeking to craft hop-heavy beers.

The substitution of hop extracts exhibited minimal impact on sensorial experience, fermentation and the volatile composition of the final product, when compared with hop pellets. While both brewing methods yielded comparable hop-derived aromatic volatiles, notable differences emerged in terms of beer haze. The lower haze levels observed in the beer brewed with hop extract were attributed to the absence of hop components available for complexation. It is important to note that even though the HEB had lower haze, it was more stable over time compared to the TPB. Therefore, hop extracts would allow brewers to create a more stable beer. In cases where increased haze levels are preferred, liquid hop-derived haze additions would be a commercially available solution.

In summary, this study underscores the potential of hop extracts as a versatile tool for crafting hop-forward beers. The similarity of physical and sensory attributes and stability under ageing conditions gives brewers the confidence to explore the application of hop extracts as a strategic means of minimizing dry-hop related beer losses while upholding beverage integrity. As the brewing landscape continues to evolve, the findings of this study provide valuable guidance for optimizing brewing processes and product development, contributing to the advancement of brewing science and the enhancement of beer quality.

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