M. Kohles, F. Gutsch, M. Zarnkow, F. Jacob, R. Novy and C. Schönberger

An approach to develop an external dry hopping method by restoring the aroma transfer through dilution

This work encompassed testing the impact of high mass concentrations of substances on the transfer of aroma and bitter compounds during dry hopping. For this purpose, a novel dynamic process for the production of cold-hopped beers was implemented on a laboratory scale. In this process, a suspension of hops and beer with a high concentration of 6.5 % w/w hops is prepared and diluted to 1.5 % w/w with beer. Then, the particulate matter is immediately separated from the beer using a filter. Evaluation of the bitter compounds, terpenes, esters and thiols using analytical methods indicated that the transfer of these compounds during dry hopping occurred similarly in samples which were hopped at the higher rate and subsequently diluted compared with those hopped at 1.5 % w/w but were not diluted. In sensory trials conducted by a panel of trained tasters (n = 10), no significant difference up to a significance of α = 0.20 could be detected by means of discriminative testing. Furthermore, no significant difference (α = 0.05) was found between the attributes of the beers in descriptive testing.

Descriptors: aroma transfer, dry hopping, dilution, extraction, hop oil, beer

1 Introduction

"One pound weight of the best hops, as taken from the pocket, should be infused into each barrel of ale," is *Herbert's* recommendation from 1872 for brewing a pale ale [1]. The process of adding hops to barrels of beer most likely represents the most traditional of the various dry hopping techniques based back to 1687 and older [2]. Based upon the original method, a wide variety of other techniques for the aqueous extraction of hops after fermentation have become established in breweries over the past 130 years [3, 4]. They all have one characteristic in common: the hops are added in the desired concentration to the respective tank where they are extracted according to the grams added per hectoliter, at the greatest possible concentration gradient for the respective aroma compounds. While hop additions of a few hundred grams per hl were still common, especially in the first half of the last century [5–7], additions of over 2.2 kg/hl are practiced today [8, 9].

However, as has been shown by *Lafontaine* [8], extraction of the desired compounds from the hops into the liquid phase is inhibited as the quantity of the hop addition increases during static dry hopping. Therefore, more and more dynamic dry hopping processes are applied in the brewing industry to increase extraction efficiency.

In this dynamic extraction techniques the extraction incorporates free convection as well as diffusion processes according to Fick's law to achieve mass transfer of these aroma compounds. In this case, fields of turbulent flow are generated by agitators or the resultant flow velocities. In contrast to static processes, turbulent flow can significantly influence and enhance the mass transfer rate. Nevertheless, the mass transfer rate decreases as the concentration of hop compounds increases [10, 11].

The primary objective in conducting these trials was to examine the impact of high mass concentrations of hops during dynamic dry hopping on the mass transfer of volatile and non-volatile aroma compounds. Furthermore, these trials were performed to determine whether the transfer rate, slowed by the high mass concentration, could be restored within a short time by reducing the mass concentration through subsequent dilution with additional liquid. This could establish new dynamic dry hopping techniques in which the hop particles no longer have to enter the fermentation tank for extraction and remain there for several days, as is the case with current dynamic techniques. The impact of this dilution on the sensory characteristics of the product was investigated as well.

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2 Material and methods

2.1 Hops and base beer

The hop variety Citra® was selected due to its widespread availability around the world and because it is frequently employed for

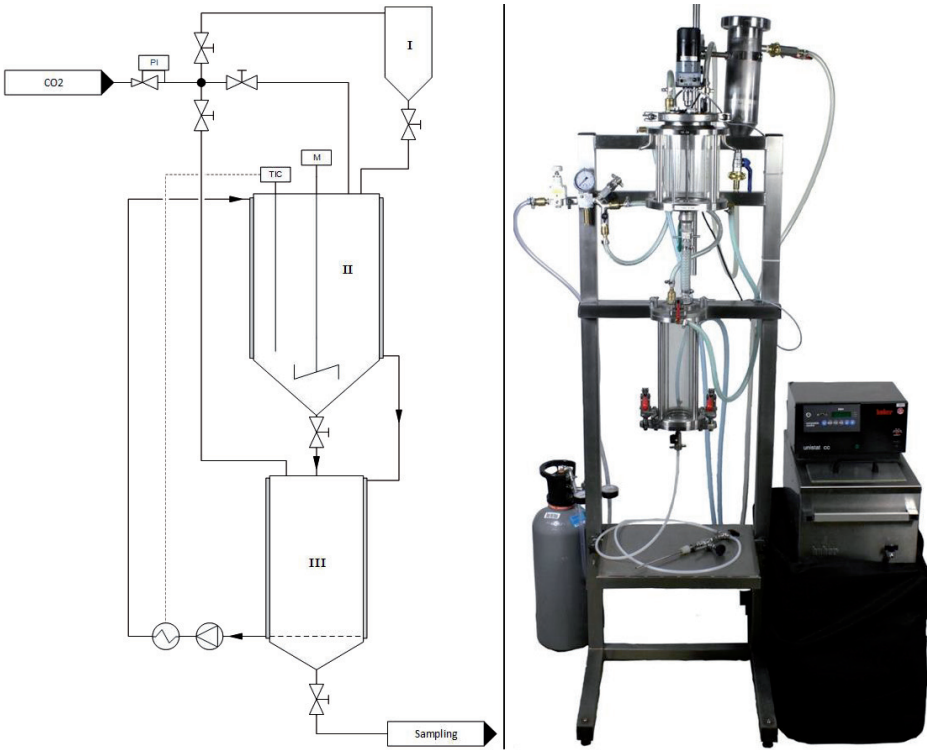

Fig. 1 **A schematic and a photograph of the device used in the experimental trials**

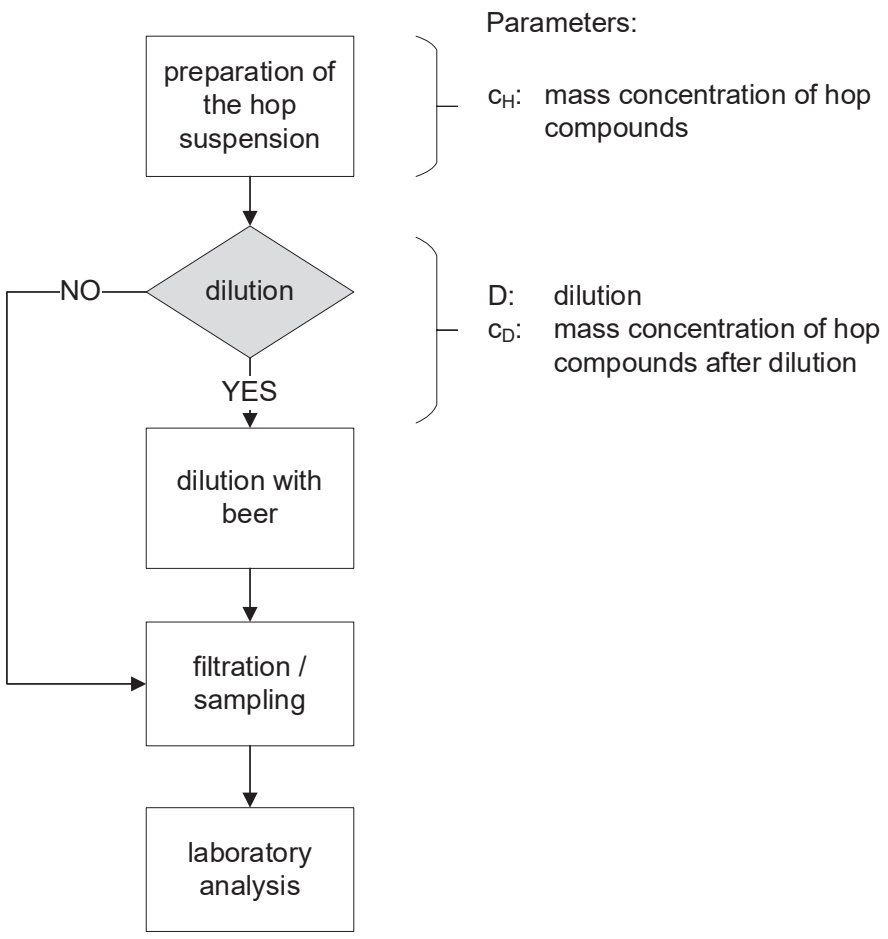

Fig. 2 **The sequence of process steps and the parameters for the experimental trials**

dry hopping. Citra® was also the most widely cultivated hop variety in the USA in 2019 [12, 13]. The used hop product in the trials was BBC Pure Hop Pellet™ (BBC).

The base beer utilized in the trials was a non pasteurized, unfiltered and non dry hopped lager beer brewed by a commercial brewery with an alcohol content of 5.3 % by volume, 14 mg/l iso-α-acids and a pH of 4,57. The beer was provided in 50-liter kegs and was stored at 4 °C until required.

2.2 Experimental setup

The trials were carried out using the device developed by the company banke GmbH (Taufkirchen, Germany), which is depicted schematically and in a photograph in figure 1.

The developed dry hopping device consisted of a dosing vessel (I), a dispersion vessel for producing the hop suspension (II) and a filtration vessel (III) for separating the particulate matter. A stainless steel slotted screen with slots 200 µm wide was employed as the filter in this vessel. Vessel I and II were equipped with a cooling jacket, the temperature was controlled using a Unistat CC thermostat (Huber, Offenburg, Germany).

A straight blade agitator with a diameter of 50 mm and a vane height of 9 mm was installed to stir the contents of the dispersion vessel (I). All of the vessels were connected to food grade CO_2.

2.3 Protocol for dry hopping

Sample preparation was conducted according to the flowchart shown in the overview in figure 2.

The required mass of beer was filled from the dosing vessel into the dispersion vessel under counterpressure and were brought to the proper temperature under constant agitation. The pellets were then added to the dispersion tank. In order to remove the oxygen introduced by adding the pellets from the headspace of the dispersion vessel, it was again purged with CO_2 and pressurized to saturation pressure. Once the hops had been added, the time allotted for agitation and dispersion of the hops throughout the medium was 120 min at a constant velocity of 50 rpm. In the trials in which the dilution was carried out, a corresponding quantity of the medium was again introduced into the

Table 1 Analysis of the hop pellets

		BBC pellets
Analyte		
cohumulone	%	2.8
n-/adhumulone	%	9.0
total humulones	%	11.7
humulinones	%	0.5
hop oil content	ml/100 g	2.04
alpha-pinene	% of total oil	< 0.1
beta-pinene	% of total oil	0.7
myrcene	% of total oil	54.2
limonene	% of total oil	0.2
cis-linalool oxide	% of total oil	< 0.1
trans-linalool oxide	% of total oil	< 0.1
beta-caryophyllene	% of total oil	7.3
linalool	% of total oil	1
alpha-terpineol	% of total oil	< 0.1
citronellol	% of total oil	0.6
nerol	% of total oil	0.1
geraniol	% of total oil	0.5
humulene	% of total oil	13
caryophyllene oxide	% of total oil	0.4
4-MMP	µg/kg	3.2
3-MH	µg/kg	< 5.0
3-MHA	µg/kg	< 0.1

dosing vessel and added to the dispersion vessel after mixing.

The contents of the dispersion vessel were transferred to the filtration vessel immediately upon completing the dilution step and filtration was started and finished within 60 s. The particulate-free filtrate flowing out of the vessel was filled directly into 0.75 l brown glass sample bottles purged with CO_2 using a counterpressure filler.

Experimental trials were conducted using the parameters listed in table 1.

2.4 Trials

Preliminary trials had already indicated that the mass transfer rate during cold hopping is increasingly inhibited as the mass concentration of hop compounds rises. One objective of this part of the experiment was to confirm this observation. For this purpose, the amount of mass transfer was compared between two dry-hopped beers with different mass concentrations of hop compounds (c_H = 6.5 % w/w and 1.5 % w/w) after removal of the particulate material through filtration.

The mass transfer of hop compounds that had been inhibited up to that point would immediately be restored when the suspension with a high mass concentration of hop compounds was diluted with beer, meaning that more beer was added. This observation was confirmed by the dilution of a hop suspension at a concentration of 6.5 % w/w to 1.5 % w/w through the addition of the standard beer, which had not been dry hopped. Immediately following the dilution, the beer was filtered, and the particulate material derived from the hops was completely separated from the beer. The dilution of the beer with the highly concentrated hop compounds, i.e., this part of the experimental trial, also served as a reference value for later experimental trials. This basic procedure was also adopted for the other trials and has therefore been defined as the reference test.

A dispersion/agitation period of 120 min was chosen to allow complete dispersion of the hop pellets into hop powder and to initiate the transfer of hop compounds to the beer. The temperature of the suspension was set at 15 °C, which corresponds to a temperature at which dry hopping is commonly carried out in breweries according to a survey from McIlmoyle et. al [14].

2.5 Treatment of the samples and composite samples

To prevent the absorption of the volatile aroma compounds into the compound liner material present on the inner side of the crown caps used as closures on the bottles [15], four layers of aluminum foil were inserted between the mouth of the bottle and the crown cap. To avoid changes in the composition of the samples, they were not stored frozen [16, 17]. Rather, the filled bottles were stored at 4 °C.

A series of tests carried out in advance indicated that the analytical results of a composite sample consisting of a mix of individual samples were within the standard deviation of each of these samples, which were also measured independent of one another. The only exception among the analytical test results, i.e., not within the standard deviation, was linalool; whereby the standard deviation for the linalool was very low. Gas chromatographic analysis of the individual samples was not carried out in order to reduce the number of samples analyzed. Instead, the samples were mixed to create a composite sample immediately prior to performing the

Table 2 List of parameters for the respective trials

Parameter		Unit	Trials		
			1.5 % w/w	6.5 % w/w (reference)	1.5 % w/w D
mass concentration of hop substances	c_H	% w/w	1.5	6.5	6.5
dilution	D	–	no		yes
mass concentration of hop substances after dilution	c_D	% w/w	–		1.5

analysis. The individual samples were measured three times using HPLC, which served as a standard for comparison.

2.6 Analytics

2.6.1 Beer analysis

The analysis of the hop bitter compounds was performed according to EBC method 9.50 [18]. The standard DCHA humulinones, ICS-Hum1 of the laboratory Veritas was used for calibration of the humulinones.

The HS-SPME-GC-MS/MS method developed by *J. Dennenlöhr* et al. was employed for analysis of the terpenes and terpenoids [19]. A gas chromatograph ("7890B gas chromatograph interfaced to a 7000C triple quadrupole mass spectrometer") manufactured by Agilent Technologies (Santa Clara, CA, USA) was used. This device was operated with a 50/30 µm DVB/CAR/PDMS fiber, and a HP-5MS UI (30 m × 0.25 mm × 0.25 µm) column and a MS/MS detector. The stable isotopes d2 myrcene, d5 linalool, and d6 citronellol were employed as standards. The detailed parameters of the method can be found in the publication by J. Dennenlöhr et al. [19].

The analysis of the esters was performed with a gas chromatograph ("Shimadzu GC 2010 interfaced with a MS-QP2010 Plus") manufactured by the Shimadzu Corporation (Kyōto, Japan). The device was equipped with a 50/30 µm DVB/CAR/PDMS fiber with a SH Rtx 5 (30 m × 0.25 mm × 0.25 µm) column and a MS detector. ^{13}C-methyl octanoate was used as the standard.

The thiols were analyzed with a gas chromatograph ("7890B gas chromatograph interfaced to a 7000C triple quadrupole mass spectrometer") manufactured by Agilent Technologies (Santa Clara, CA, USA). This was equipped with a 65 µm PDMS/DVB fiber and an additional derivatization was carried out on the SPME fiber. The used method was presented by *Nils Rettberg* et. al. in the year 2018 at the EBC Symposium "Recent Advances in Hop Science." [20]

2.6.2 Sensory analysis

In addition to laboratory analysis, the samples were also evaluated sensorially. Both a discriminative test and a descriptive tasting of the non-diluted 1.5 % w/w and the diluted 1.5 % w/w D (reference) samples were performed.

For the discriminative tests, a triangle test according to DIN EN ISO 4120:2007-10 [21, 23] and a descriptive tasting according to the Hopsessed® evaluation scheme of BarthHaas, Nuremberg, were employed [22]. For the descriptive tasting hop aromas were divided into 12 general categories and evaluated on a scale from 0 to 10.

In addition to the characteristics of the aromas and flavors, the panel members (n = 10) assessed six attributes used to distinguish the quality of the hops in beer.

The tasters were also asked to estimate the perceived bitterness. Furthermore, as noted by *Algazzali, V.* and *Shellhammer, T.*, bitterness was calculated from the analysis results for iso-α-acids in mg/l and humulinones in mg/l (66 % of iso-α-acids) [24].

2.6.3 Statistical analysis

Statistical analysis was performed using OriginPro 2020b (OriginLab, Massachusetts). For descriptive statistics, the standard deviation was calculated for the HPLC results and presented as error bars in the graphs.

For the HPLC analysis, a single-factor ANOVA ($p > 0.05$) and post hoc Bonferroni test were also calculated to determine if there was a significant variance in the differences between the values for the mean at a level of significance of 0.05.

The Shapiro-Wilk test ($p > 0.05$) was used to evaluate the samples for normal distribution and Levene's test ($p > 0.05$) was employed to check for variance homogeneity.

Table 3 Analysis results for bitter substances and pH

		Base beer	Reference		
Analyte			**1.5 % w/w D**	**1.5 % w/w**	**6.5 % w/w**
bitter compounds:					
iso-α-acid content	mg/l	14.43 ± 0.29	9.50 ± 0.33	11.37 ± 1.11	5.60 ± 0.43
mean change in iso-alpha-acids	mg/l	–	– 4.93	– 3.07	– 8.83
mean change in iso-alpha-acids	100 g/hl	–	– 0.33	– 0.20	– 0.14
cohumulone	mg/l	< 0.4	10.33 ± 0.12	9.57 ± 0.41	19.17 ± 0.45
n-/adhumulone	mg/l	< 0.7	16.43 ± 0.17	14.80 ± 0.45	35.47 ± 0.99
total humulones	mg/l	< 1.1	26.77 ± 0.25	24.37 ± 0.45	54.70 ± 1.39
humulinones	mg/l	1.67 ± 0.21	42.43 ± 0.84	44.73 ± 0.19	109.93 ± 0.94
pH values:					
measured pH value (± SD)	pH	4.57 ± 0.00	4.78 ± 0.01	4.79 ± 0.01	5.00 ± 0.01
mean pH change	pH	–	0.20	0.21	0.43
mean change per 100 g/hl	pH	–	0.014	0.014	0.007

3 Experimental trials

3.1 The effect of dilution on hop compounds

3.1.1 Hop bitter compounds

The data in table 3 show that dry hopping on the cold side of production resulted in a decrease in the concentration of iso-α-acids in all of the trials, this effect was shown by *Maye* et. al. in 2016 [25].

The iso-α-acid content in the 6.5 % w/w sample drops to a greater extent, to 8.83 mg/l, compared with the 1.5 % w/w D reference sample with 4.93 mg/l, and in 1.5 % w/w sample with 3.07 mg/l. This is due to the fact that approximately four times the quantity of hop material was added to the suspension in the 6.5 % w/w sample. Since iso-α-acids exhibit a low solubility in water and display surface-active properties, a larger amount of iso-α-acids was separated from the suspension together with the hop solids due to the presence of more plant material. The drop in iso-α-acids in the highly concentrated 6.5 % w/w sample is the lowest of the three samples. This value is 0.14, expressed in terms of 100 g of hop pellets added per hectoliter.

The data in table 3 show an increase in the humulones and humulinones for all samples compared to the original concentrations measured in the base beer, this was also shown by Maye et. al in 2016 [25].

In the base beer, no measurable concentration of humulones could be detected and only a small amount of humulinones, at 1.67 mg/l. This is due to the low solubility of humulones at low pH levels [26].

A clear difference exists between the 6.5 % w/w sample and the 1.5 % w/w D sample. This also holds true for the comparison between the 6.5 % w/w and the 1.5 % w/w samples.

However, no significant difference could be detected for any analyte between the 1.5 % w/w and the 1.5 % w/w D samples.

This means that only the quantity of dry hops in the hop addition has an impact on the bitter compounds, i.e. on the concentrations of iso-α-acids, humulones and humulinones, while dilution from 6.5 % w/w to 1.5 % w/w has no effect.

3.1.2 pH

The addition of hops resulted in an increase in pH in all of the trials (refer to Table 3). In the two 1.5 % w/w samples, with and without dilution, an identical, absolute pH increase of 0.2 was measured, which amounts to 0.014 per 100 g/hl. The pH increase in the 6.5 % w/w sample was about twice as high at 0.43, but this also corresponds to a specific increase in the pH of just 0.007 per 100 g/hl.

In essence, there are no significant differences between the two techniques, regardless of whether the pH changes.

3.1.3 Terpenes

Overall, significant variations between the samples were observed in the measurement of the terpenes. This can be attributed to the high volatility of most terpenes and the complex analytical procedures involved in their determination. In general, the concentrations of terpenes in the 6.5 % w/w samples are higher than those in the two 1.5 % w/w samples. However, it should be noted that the concentration of terpenes does not increase proportionally with a higher hop addition.

This suggests that phenomena occur at high hop concentrations which inhibit the mass transfer of aroma compounds into the beer medium [8].

The terpene concentrations of the three samples are shown in table 3. The caryophyllenes and humulenes do not appear to be impacted by the inhibiting effects on mass transfer. Their concentrations in the 6.5 % w/w sample of 82.4 µg/l and 208.5 µg/l, respectively, are significantly higher than those found in the 1.5 % w/w samples. S. Lafontaine and T. Shellhammer were also able to measure this effect in static cold hopping tests [8].

Comparison of the 1.5 % w/w samples with the 1.5 % w/w D samples makes it clear that dilution does not seem to be associated with any disadvantages regarding the yield of the terpenes. Especially for linalool at concentrations of 1389.9 µg/l and 1378.8 µg/l as well as myrcene at 544.8 µg/l and 443.2 µg/l but also for beta pinene at 11.5 µg/l and 10.6 µg/l. The amounts measured are very similar if the standard measurement errors of the analysis methods are taken into account. The exception is geraniol, with a concentration of 608.4 µg/l measured in the 1.5 % w/w D sample reaching a similar value of 710.3 µg/l in the 6.5 % w/w sample. By contrast, the geraniol concentration is only 172.2 µg/l in the 1.5 % w/w sample.

However, the differences between the two 1.5 % w/w samples are small if the total sum of the terpenes is considered. It is possible that the differences have arisen through variations in sample preparation and in the subsequent analysis.

3.1.4 Esters

The esters, listed in table 3 follow a pattern similar to that of the terpenes. The values for the 1.5 % w/w D sample and the 1.5 % w/w sample lie within a comparable range. However, differences were observed between the concentrations of the isobutyl isobutyrate and ethyl isobutyrate (155.2 µg/l and 24.3 µg/l, respectively) in the 1.5 % w/w sample, compared to the same esters in the 1.5 % w/w D sample (106.7 µg/l isobutyl isobutyrate and 15.5 µg/l ethyl isobutyrate).

The measured values were higher for all of the esters in the 6.5 % w/w sample, but they do not reflect the much larger addition of more than four times the amount of hops. Therefore, as observed with the terpenes, the mass transfer of the hop compounds into the beer matrix appears to be inhibited at high ester concentrations.

Table 4 Analysis results for hop aroma compounds

		Base beer	Reference		
Analyte			**1.5 % w/w D**	**1.5 % w/w**	**6.5 % w/w**
Terpenes:					
alpha-pinene	µg/l	0	< 1.0	< 1.0	< 1.0
beta-pinene	µg/l	< 1.0	10.6	11.5	22.7
myrcene	µg/l	2.6	443.2	544.8	1541.3
limonene	µg/l	< 1.0	20.6	71.6	113.9
cis-linalool oxide	µg/l	1.1	44.0	< 1.0	< 1.0
trans-linalool oxide	µg/l	1.0	39.2	67.1	140.9
linalool	µg/l	12.0	1378.8	1389.9	2291.9
alpha-terpineol	µg/l	5.6	25.7	38.8	17.9
citronellol	µg/l	7.2	22.0	16.6	68.8
nerol	µg/l	2.0	76.5	45.7	95.4
geraniol	µg/l	5.4	608.4	172.2	710.3
caryophyllene	µg/l	< 1.0	10.1	13.9	82.4
humulene	µg/l	< 1.0	24.7	36.6	208.5
caryophyllene oxide	µg/l	< 1.0	11.0	< 1.0	< 1.0
∑ ***without myrcene**	µg/l	34.3	2271.6	1863.9	3752.7
∑ **total**	µg/l	36.9	2714.8	2408.7	5294.0
Esters:					
ethyl isobutyrate	µg/l	4.3	15.5	24.3	34.8
2-methylbutyl isobutyrate	µg/l	< 1.0	46.4	36.5	68.6
methyl 2-methylbutyrate	µg/l	1.4	2.8	< 2.0	2.5
isobutyl isobutyrate	µg/l	1.2	106.7	155.2	167.2
propyl 2-methylbutyrate	µg/l	< 1.0	< 1.0	< 2.0	< 2.0
butyl isobutyrate	µg/l	1.3	6.0	4.2	4.4
ethyl 4-methylpentanoate	µg/l	< 1.0	3.2	2.8	2.9
2-methylbutyl-2-methylbutyrate	µg/l	< 1.0	5.3	4.7	4.1
2-methylbutyl isovalerate	µg/l	1.2	9.0	7.7	8.8
methyl geranate	µg/l	3.2	260.8	190.4	290.3
geranyl propionate	µg/l	< 1.0	7.1	2.5	7.9
geranyl acetate	µg/l	< 1.0	< 1.0	< 1.0	< 1.0
phenethyl isovalerate	µg/l	< 1.0	< 1.0	< 1.0	< 1.0
geranyl isobutyrate	µg/l	< 1.0	29.9	13.1	39.0
∑ **total**	µg/l	12.6	492.7	441.4	630.5
Thiols:					
4-MMP	ng/l	< 1.0	55.0	22.2	163.9
3-MH	ng/l	10.8	18.9	30.7	59.2
∑ **total**	ng/l	10.8	73.9	52.9	223.1

There is, however, little difference in the sum of total esters between the two samples.

3.1.5 Thiols

The values for 4-MMP and 3-MH obtained for the 6.5 % w/w sample were significantly higher than the measured values of the 1.5 % w/w D and the 1.5 % w/w samples (refer to Table 4). It appears that the higher thiol concentrations roughly correspond to the larger hop addition. Therefore, unlike the terpenes and esters, inhibition of the mass transfer of the thiols did not occur at these higher hop concentrations.

Among the 1.5 % w/w samples, the 1.5 % w/w D sample with a concentration of 55.0 ng/l contains higher amounts of 4-MMP than the 1.5 % w/w sample at 22.2 ng/l. With regard to 3-MH, the concentration is slightly higher at 30.7 ng/l in the 1.5 % w/w sample. As already discussed in the analysis of the individual samples in

section 2.5, the analysis methods employed in the determination of thiols are subject to a large standard deviation.

The total sums of the thiols are within a close range of one another at 52.9 ng/l and 73.9 ng/l and point to only a small difference among the 1.5 % w/w samples if the standard deviation for the analysis method is taken into consideration.

3.1.6 Transfer rates

The transfer rates for linalool of 52 % and 53 % for the two 1.5 % w/w samples are almost the same as those for linalool from hop pellets into the cold, dry-hopped beer. By contrast, the 6.5 % w/w sample exhibits a considerably lower yield of 20 %. This confirms that the percentage of mass transfer is considerably lower with increasing mass concentration. This effect can also be observed for nerol and humulinones.

No clear results were obtained with regard to limonene, geraniol and nerol.

3.2 Sensory analysis

3.2.1 Discriminative testing

The test showed no significant difference between the two production methods. Five samples were correctly identified up to a level of $\alpha = 0.20$. This does not unequivocally confirm the similarity of the two samples, but it does indicate a tendency in a certain direction, which was corroborated by the descriptive tasting results described in section 3.2.2 .

3.2.2 Descriptive testing

The descriptive taste test showed that the evaluation results from well-trained tasters were very similar within each category. The results for the 1.5 % w/w D sample and the 1.5 % w/w sample are displayed in the form of a spider diagram in figure 3.

At a significance level of 0.05, a paired, two-sample t-test failed to reveal a significant difference between the attributes assessed. Thus, this confirms the results of the discriminative test, namely that no perceptible difference exists between the two samples.

The perceived bitterness of the 1.5 % w/w D sample at 35.09 ± 5.19 bittering units (BU), exhibits a difference of only 0.54 BU from the 1.5 % w/w sample with a value of 34.55 ± 4.59 BU. At a significance level of $\alpha = 0.05$, there is no significant difference in perceived bitterness between the two samples. The calculated perceived bitterness of 34.46 BU for the 1.5 % w/w D sample and 38.08 BU for the 1.5 % w/w sample lie within the standard deviation for the beers evaluated through sensory analysis. Therefore, this indicates that the values for iso-α-acids and humulinones can

Table 5 Transfer rates for selected hop compounds

		Linalool	Limonene	Geraniol	Nerol	Humulinones	4-MMP
1.5 % w/w	%	53	14	13	17	57	46
1.5 % w/w D	%	52	4	44	28	54	115
6.5 % w/w	%	20	5	12	8	33	79

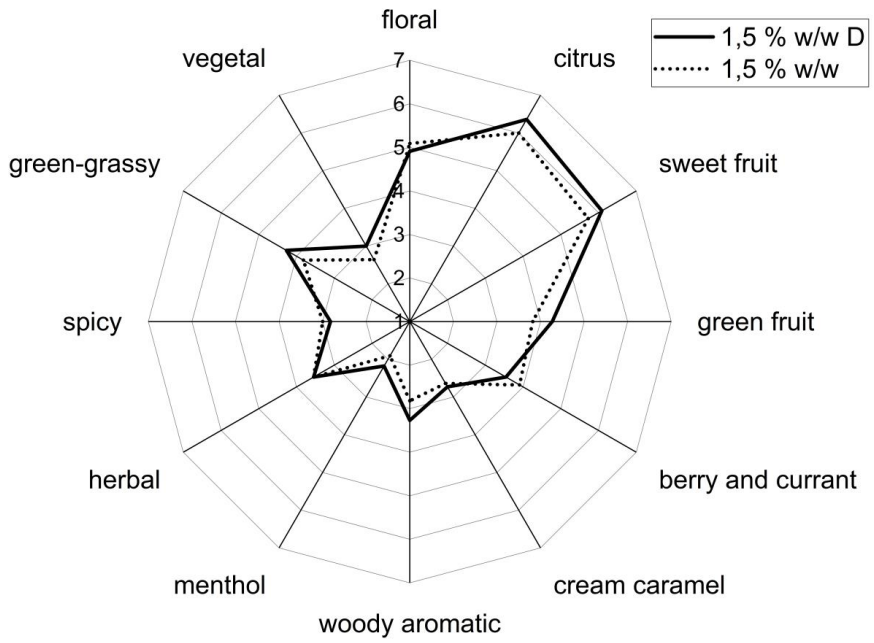

Fig. 3 Sensory analysis results for hop aroma intensity

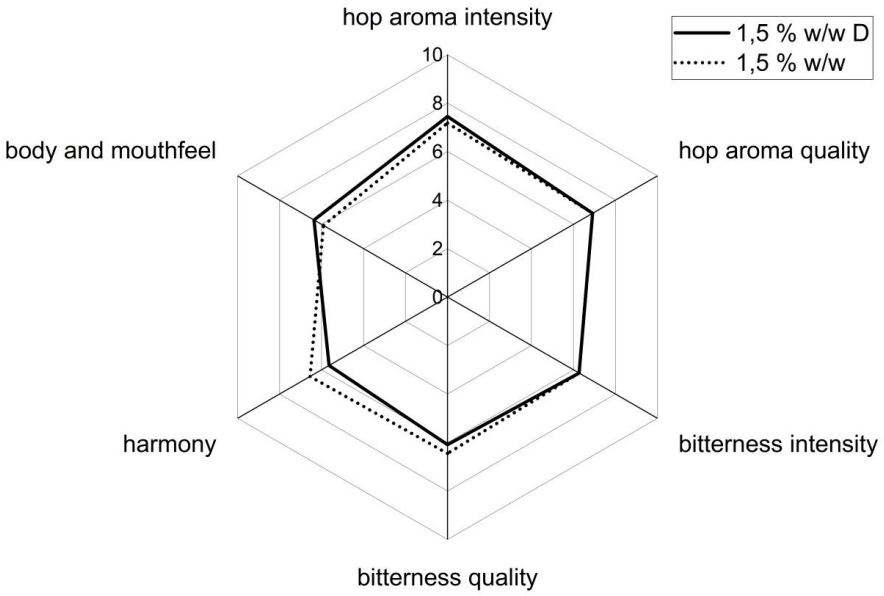

Fig. 4 Sensory analysis results for bitterness

serve as a good estimate of the perceived bitterness in the beer.

3.2.3 Evaluating bitterness

Figure 4 shows that the assessment of bitterness intensity and hop aroma quality were nearly identical.

Of the samples evaluated, only the 1.5 % w/w sample received higher ratings for the sensory attribute of harmony. Nevertheless, no significant difference between the two samples was found for any of the attributes at a level of significance 0.05. The results for the discriminative test also confirm that no significant difference exists between the two production methods.

4 Discussion and summary of the results

The goal of this research was to clarify whether dry hopping can be carried out externally by creating a suspension of hop products typically used for dry hopping, subsequently diluting the suspension and removing the particulate hop material immediately without the loss of any valuable compounds. For this purpose a laboratory scale setup was developed to simulate the external dry hoping. This setup delivered results with a high degree of reproducibility across all of the tests performed in this trial.

The findings in this study confirm prior observations made as part of the preparations for these trials and past research on this topic, namely that the rate of aroma transfer decreases when large amounts of hop pellets are added during dry hopping. Hops added to beer in excess of four times the regular quantity for dry hopping do not result in a concentration that is four times higher for most of the aroma compounds in the beer. One exception are the thiols, especially 4-MMP and 3-MH, as well as caryophyllenes and humulenes. No reduction in the transfer of these aroma compounds into the beer could be determined.

After 120 min of dispersion and agitation, the dry-hopped beer with a mass concentration of 6.5 % w/w was diluted. The mass transfer rates for both the bitter compounds and the aroma compounds were immediately restored within a very short contact time at a mass concentration of 1.5 % w/w after dilution. This shows that the mass transfer of compounds derived from hops can be very quickly restored once the inhibiting conditions have been lifted, i.e., by lowering the concentration of hop substances in the medium.

The sensory analysis results obtained from the taste tests mirror the analytical data. No perceptible difference was observed between the undiluted beers and the diluted beers in the discriminative and descriptive tests.

This phenomenon offers entirely new options for the day-to-day dry hopping techniques performed in breweries. The initial dispersion/suspension step allows the hop pellets to disintegrate into hop powder, to absorb beer and to expand in size. Although maximum efficiency is not achieved for the extraction in this step, it can be compensated through the subsequent dilution of the suspension. The time required for dilution, so that complete extraction can be achieved, is very short at around 60 seconds.

These rapid extraction times enable brewers to extract hops efficiently outside of the maturation tank and to reduce the contact time between hops and beer – conventionally over a period of several days – to a mere few hours. Tank occupancy time in the brewery can also be significantly reduced through application of these techniques. A change in the biotransformation of the hop aromas by the yeast contained in the beer is not to be expected, since the aroma components are fully available to the yeast cell as in previous dynamic dry hopping processes. On the other hand, it is feasible that the enzymes responsible for the hop creep effect are largely removed with the plant material, which could reduce this effect.

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