DRY HOPPING – A STUDY OF VARIOUS PARAMETERS

Consequences of the Applied Dosing Method

This supplement to the publication "Revival of a Process (Dry Hopping – Basics and Techniques)" [1] featured in this year's March edition of BREWING AND BEVERAGE INDUSTRY INTERNATIONAL deals with the presentation of various parameters of cold hopping, also referred to as dry hopping. These include dosing quantities, temperature, dosage method, and contact times, all as tested on laboratory and pilot plant scale.

Analysis

All the hop analyses were carried out in the Hopsteiner-HHV laboratory, using the conductometric EBC 7.5 and EBC 7.7 HPLC (Standard ICE 3) methods as well as the EBC 7.10 and 7.12 methods for the aroma substances.

The determination of the myrcene content of the beers was carried out at Weihenstephan's Research Centre for Brewing and Food Quality using the MEBAK III 1.4 method.

All other beer analyses were carried out in our own laboratory. The determination of alpha acid in beer using HPLC is based on our own method, using the ICE 3 Standard. Similarly, the gaschromatographic analysis used to determine the linalool content is our own in-house method.

Table 1: Absolute Increases after Dry Hopping

Hop Variety	Alpha Acid mg/l	Myrcene µg/l	Linalool µg/l
German Magnum	2.6	9.0	12.1
German Herkules	5.9	20.0	12.6
US Apollo	6.6	28.0	21.2
US Bravo	0.6	4.0	27.4
US Calypso	3.2	13.0	20.6
US Delta	1.6	1.0	25.4
NZ Nelson Sauvin	5.4	29.0	21.1
Average	3.7	14.9	20.1

Dry Hopping Trials in Kegs

Initial dry hopping trials were carried out by simply putting hops into kegs prior to filling. The main objective was to observe aroma and flavour changes in the beer, but, at the same time, we acquired data about the solubility of the main hop-related ingredients.

In the first series of trials, for which sensory results have already been published, small quantities of pellets type 90 (50 g/hl) were added to the keg after the main fermentation. The finished beer was filtered after two weeks of contact time [2, 3]. The increases in alpha acids as well as the two aroma substances myrcene and linalool are shown in table 1. No change in iso-alpha acid content is registered as none is added (not shown in table 1). On another note, due to its poor solubility in beer, the alpha acid content increases on average by only 3.7 mg per litre or approximately 6 per cent.

The very unpolar terpene myrcene has an even lower yield of less than 1 per cent. Linalool, on the other hand, which is a terpene alcohol, dissolves far more easily than myrcene. The very low linalool content in hops means that it is dosed in very small quantities. The effect of both myrcene and linalool is easily recognisable as a result of dry hopping, so we

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decided to focus all further investigation only on linalool as it is easier to analyse than myrcene.

Consequently the analysis of aroma substances for all further trials is limited to this substance and also to the non-isomerised alpha acid which, despite being far less flavour intense than iso-alpha acid, is nevertheless a good indicator to balance the effects of dry hopping dosage rates. Alpha acid is also a major contributor to beer foam stability [4].

In a further series of trials the hops were dosed based on their total oil content rather than the quantity of hops. This was done using 2.0 grams total oil per hl. The beers were India Pale Ales (IPAs), albeit modestly hopped for this type of beer. They were brewed at the Research Brewery of Lehrstuhl für Brau- und Getränketechnologie, TU München-Weihenstephan.

Table 2 shows the analytical data of the original beer prior to dry hopping, which was filled into kegs. Prior to filling, several hop varieties were added to the kegs in powder form. Among the hops used were three of the recently registered, new Hüll varieties (Polaris, Hallertau Blanc, Mandarina Bavaria), two as yet unregistered varieties bred in Hüll (nos. 2008/020/004, 2009/001/718) and two newly bred Hopsteiner varieties from the USA (Bravo, planted in the Hallertau and the as yet unregistered variety 04190).

Based on the addition of 2.0 grams total oil per hectolitre, considerably higher quantities of hops were dosed in comparison with the above-mentioned series of trials. This is demonstrated together with the corresponding alpha acid and linalool concentrations in table 3.

Compared with the first series of trials, especially large increases of linalool are evident (see table 4), although it is important to take into consideration the fact that other varieties were used and, consequently, a direct comparison cannot be made. The rate of increase in alpha acid is similar. This means that the higher the dosage, the lower the resulting yield will be. In this case the first and second series of trials were performed in different conditions, so we decided to carry out a more detailed, laboratory scale study of the subject.

Table 2: Standard Beer Used for Dry Hopping

Alcohol Content	5.6 vol.-%
Bitter Units	32.0 BU
Iso-Alpha Acids	29.0 mg/l
Alpha Acids	3.3 mg/l
Linalool	19.5 µg/l

Table 3: Quantities and Analyses of Several Varieties for Dry Hopping

Hop Variety	Hops g/hl	% Alpha Acids HPLC	Linalool µg/g
2008/20/004	170	9.8	31.2
Polaris	70	19.6	25.6
2009/001/718	170	8.6	5.2
Hallertau Blanc	186	10.8	28.8
Mandarina Bavaria	160	9.1	22.4
Bravo	130	18.1	40.8
US 04190	556	2.7	9.6

Laboratory Scale Trials to Examine various Parameters

One litre scale trials were made in order to determine the influence of the quantity dosed, the temperature and method of addition. Of course we would not expect the results of these trials to be identically re-produced in full-scale production, but they are intended to at least demonstrate the tendencies and give tips for ensuing full-scale trials.

Table 4: Increases in Beer after Dry Hopping

Hop Variety	Alpha Acids mg/l	Linalool µg/l
2008/20/004	2.8	49.2
Polaris	2.0	23.0
2009/001/718	3.8	66.7
Hallertau Blanc	3.6	48.6
Mandarina Bavaria	4.8	50.8
Bravo	5.5	60.0
US 04190	2.8	89.4
Average	3.6	55.4

Table 5: Analysis Data for Tradition for the Laboratory Trials

LCV (EBC 7.5)	6.6 %
Alpha Acids (EBC 7.7)	5.7 %
Total Oil (EBC 7.10)	6.0 ml/100g
Linalool (EBC 7.12)	1.0 %-rel.

Table 6: Standard Beer used for the Laboratory Trials

Specific Gravity	12.4 %
Alcohol Content	5.6 vol.-%
Bitter Units	32.3 BE
Iso-alpha Acids	31.0 mg/l
Alpha Acids	0.8 mg/l
Linalool	37.0 µg/l

All the dry hopping was done using Tradition pellets type 90, for which the most important analysis data can be found in table 5. Dry hopping was not done using an ale type beer, which would have been more appropriate from a flavour viewpoint. Instead, a typically hopped Pilsner beer was used (see table 6), in order to test the solubility of alpha acid and linalool in a beer with relatively high contents of bitter and aroma substances.

Influence of the Quantity of Pellets Used

Similarly, the quantity of pellets used for this trial was calculated on the basis of the amount of total oil added. Trials were made using 1.5/2.0/2.5/3.0 g total oil per hl.

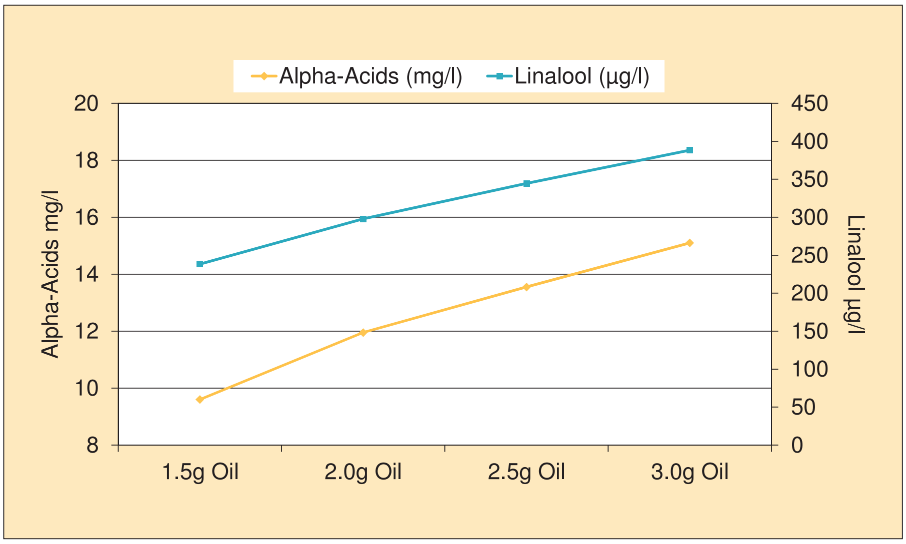

Fig. 1: Influence of the Dosed Quantity on the Solubility of Alpha Acid and Linalool

at 20 °C. Their decrease during 2 weeks of storage is within the normal range of fluctuation and can most probably not be interpreted as a reduction.

Surprisingly, at least for the laboratory trial, temperature influence on the solubility of linalool is minimal, as shown in figure 3. The dissolving process is more or less complete just one day after dry hopping. This is due to these aroma components being more polar than alpha acids.

Influence of Dosing Method

Dry hopping with pellets or leaf hops in the storage tank results in an addition of solids which

Figure 1 shows that increased dosage does not result in a linear increase of alpha acid and linalool, but, as would be expected, rather flattens out and consequently reconfirms the results of the above-mentioned trials in kegs. The higher the hop dosage, the less efficient the dissolving process.

Influence of the Beer Temperature

These trials were done using a higher dose of 2.5 g total oil per hl, in order to help recognise any temperature influence better. The numbering used in figures 2 and 3 refers to the time when the beer analysis was made:

1. Before dry hopping
2. One day after dry hopping
3. Two days after dry hopping
4. Two weeks after dry hopping

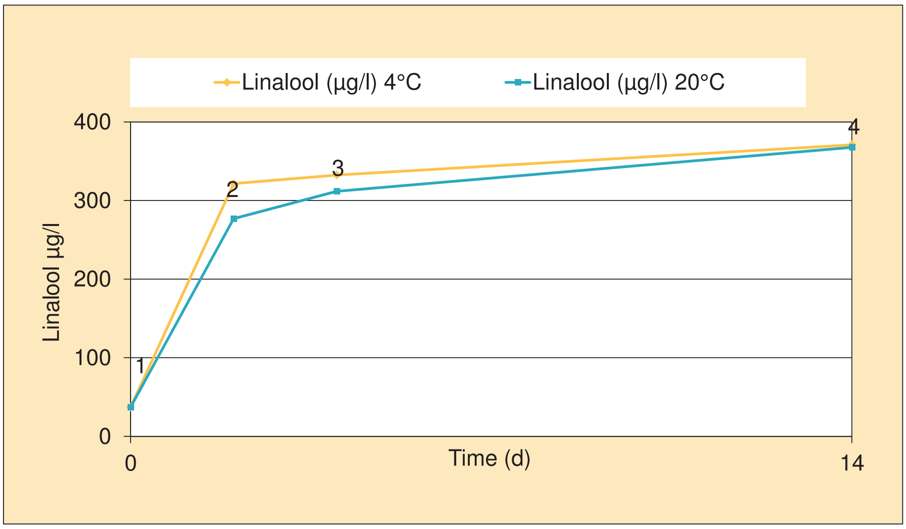

Fig. 3: Influence of Beer Temperature on the Solubility of Linalool

As post-fermentation temperatures can vary greatly in practice, extremes of 4 °C and 20 °C were selected. As would be expected alpha acids tend to dissolve quicker

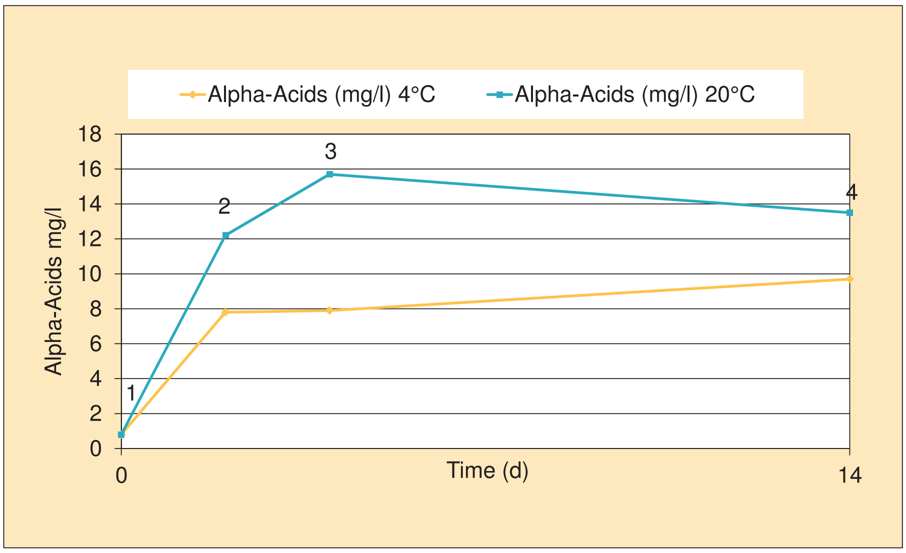

Fig. 2: Influence of Beer Temperature on the Solubility of Alpha Acid

can lead to problems during beer clarification and, if applicable, also during filtering. Very often it will be necessary to centrifuge the beer twice in order to avoid any problems during filtration. Even if the beer is pulled unfiltered, one would generally want a low solid content.

In practice, this problem is frequently dealt with by adding the hops to the conditioning tank in a finely woven sack. A laboratory trial was carried out to ascertain whether this technique worsens the solubility of the hop ingredients. A quantity of pellets corresponding to 3.0 g total oil per hectolitre was dosed to the green beer. The first quantity was added in loose form, the second in a finely woven sack and both were tested for a contact time of two weeks at 10 °C.

Figure 4 shows the varying behaviour of alpha acids and linalool. The alpha acids dissolve far more

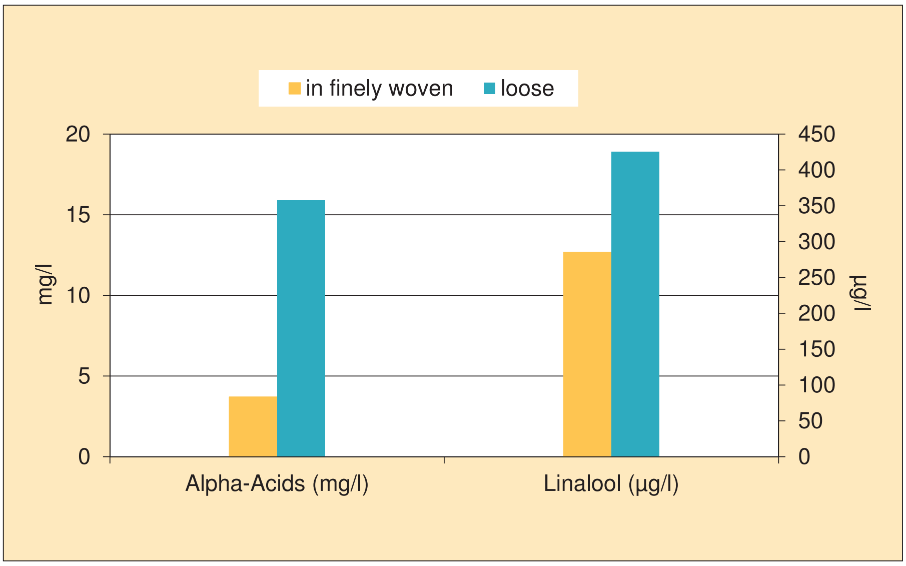

Fig. 4: Influence of the Dry Hopping Method

quickly when added in loose form, although, in practice, this is not a decisive factor. The behaviour of the aroma substances is far more important. In the case of linalool, the use of pellets in loose form gives almost 50 per cent more extraction efficiency and, consequently, the likelihood of a beer with a more intense aroma.

Trials on Laboratory Scale to Determine the Required Contact Time

Trials were done in the pilot plant to determine the contact time required in beer for both leaf hops and pellets. The beer used for this purpose was again provided by Research Brewery of Lehrstuhl für Brau- und Getränketechnologie, TU München-Weihenstephan. The beer parameters are described in table 2.

The leaf hops and the pellets used for the trials were from the same processing run. The resulting quantity yield was calculated on the basis of the quantity used. Consequently a larger quantity of leaf hops than pellets was added, but the quantity of ingredients was the same for both products (see table 7). The dry-hopped beer was tracked analytically for 18 days. Storage temperature remained at a constant 4 °C.

Figure 5 shows the behaviour of alpha acids for both leaf hops and pellets. Points of analysis are displayed in the graph as follows:

1. Before dry hopping
2. 2 hours after dry hopping
3. After 4 hours
4. After 8 hours
5. After 1 day
6. After 5 days
7. After 7 days
8. After 11 days
9. After 13 days
10. After 18 days

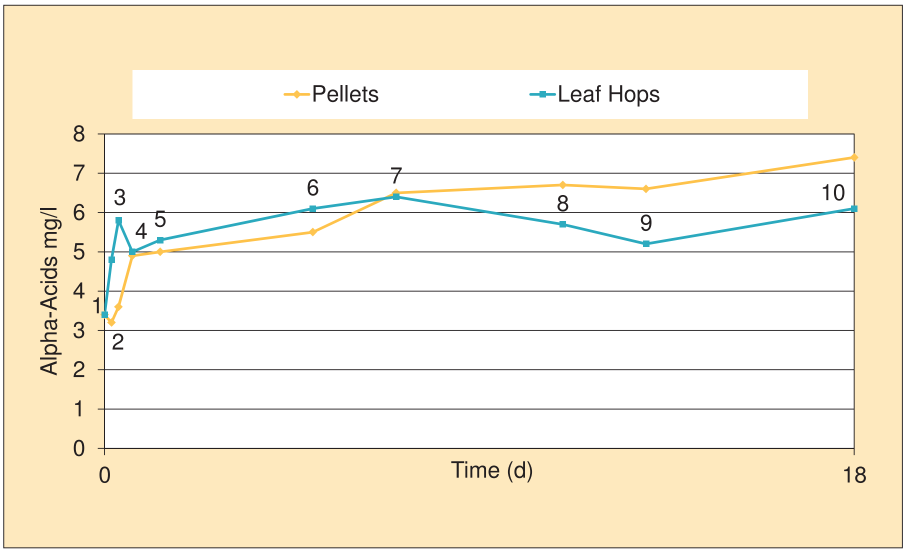

Fig. 5: Increase of Alpha Acids during Cold Hopping; Comparison between Leaf and Pellets

On the first day the alpha acids increase quickly by about 2 mg/l. However, by the 18th day their total concentration only increases by a further 1 to 2 mg/l (see fig. 5).

Table 7: Analysis Data for the Variety Tradition for the Laboratory Trials

Analysis Data	Leaf Hops	Pellets
LCV (EBC 7.5)	6.3 %	6.6 %
Alpha Acids (EBC 7.7)	5.4 %	5.7 %
Total Oil (EBC 7.10)	0.5 ml/100g	0.6 ml/100g
Linalool (EBC 7.12)	1.1%-rel.	1.0 %-rel.

There is no significant difference between leaf hops and pellets. On the other hand, there is a clear difference when comparing the behaviour of linalool in the two products (see fig. 6). A considerably higher concentration results with pellets compared to leaf hops. It dissolves relatively quickly with 50 per cent of the total acquired quantity dissolving within only one day of the total 18-day period.

When using leaf hops, two thirds of the final concentration are acquired within the first day (see no. 5). However, the final linalool level is only about 50 per cent as high as that of the pellet beer. The decisive dissolving processes for both hop products were completed within one week (see no. 7).

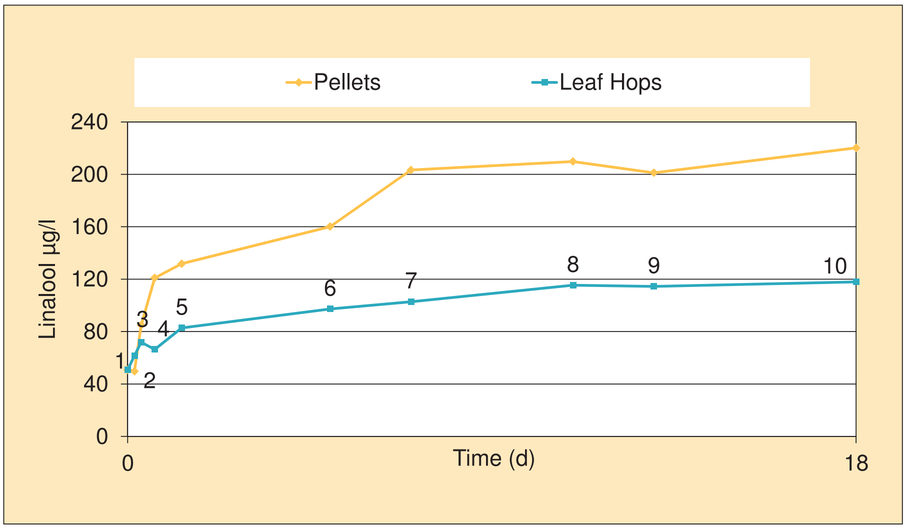

Fig. 6: Increase of Linalool during Cold Hopping; Comparison between Leaf and Pellets

Conclusion

In laboratory and pilot plant trials, various parameters which might influence the solubility of alpha acids and linalool in green beer were examined. In some cases, depending on how much was added, a significant quantity transferred into the beer, albeit not proportionally. Beer storage temperatures influence the alpha acids significantly, but hardly affect linalool contents at all.

Nevertheless, the method of hop addition appears to have a clear impact. If pellets are added to the beer in a finely woven, mesh-like container, then the solubility of alpha acid is particularly restricted. Similarly the linalool concentration only attains a considerably lower level.

Investigation into the contact time required between hops and beer shows that after one week under these conditions there were hardly any changes of concentration. Of course, these statements apply only to alpha acids and linalool and it is quite probable that some of the numerous hop ingredients might behave differently.

Basically these small-scale trials are intended to provide tips and tools which may help when implemented in practice. ☐

Hopsteiner at the drinktec: Hall B1, Stand 524

Literature

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