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Preliminary communication

HOP PELLETS TYPE 90: INFLUENCE OF MANUFACTURE AND STORAGE ON LOSSES OF α-ACIDS

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Hop pellets type 90 are the most frequent hop products used in brewing. They are usually manufactured by drying hop cones, temporary storage of dry and pressed cones, milling and pelletising. Other possibility is a complete integration of hop harvest, cone drying, milling and pelletising in continuous process without temporary storage of pressed cones. The paper deals with the losses of bitter substances (primary α-acids) during hop cones drying, their pelletisation by two manufacturing procedures and storage in different conditions. The results of this study show that integrated procedure of hop pelletising decreases the total loss of α-acids compared to the usual procedure. During storage of hop pellets (produced by integrated procedure) the highest decrease (loss) of α-acids content was observed in pellets stored at 21 °C in the presence of air and the lowest under inert atmosphere (N_2) at 4 to 7 °C, respectively.

Keywords: hop pellets type 90, manufacture, storage, α-acids loss

The fact is that hop quality is diminished during harvesting and processing into hop pellets (FORSTER, 2001a). The usual (standard) processing of hop cones into hop pellets type 90 is basically a three-step procedure: drying, temporary storage of dried hop cones (pressed into hop bales) and pelletisation. However, each step could be critical in the quality chain of hop pellets if the temperatures are increased. Higher temperature speeds up the oxidation reactions which cause degradation of bitter and aromatic hop substances and consequently decrease the brewing value of hop pellets. The basic principle of hop chemical compounds degradation primarily means the oxidation of α-acids when they are exposed to air, particularly at high temperatures during kilning,

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conditioning, pressing, temporary storage, pelletisation, transportation to a brewery and storage in brewery warehouse (WEBER et al., 1979; FORSTER, 1999; 2001a; b; 2002; 2003a; b; ROSSBAUER & MÜNSTERER, 2003; VIRANT & MAJER, 2003). Unfortunately, sometimes it is difficult, expensive and even impossible to decrease the air temperature during the critical steps of hop quality chain, but it is possible to reduce the time of hop exposure to high temperatures. Therefore, the integrated pelletisation of hop cones into hop pellets type 90 was developed (SREČEC et al., 2005). It means that all well known technical solutions are linked into an integrated system which links harvesting, kilning and conditioning or just cooling of dried hop cones with grounding, pelletisation and packing in 3-ply Al-foil bags under inert N_2 atmosphere. Nevertheless, it is well known that even this kind of packing cannot prevent degradation of α-acids when exposed to high temperatures during transportation and storage. Warm conditions over 25 to 30 °C should be avoided, because the formation of gas within the package (due to chemical reactions) can cause the rupture of foil. This inevitably leads to oxidation or even to the total spoilage of the pellets. Namely, increase of temperature causes increase of gas volume inside of 3-ply Al-foil bags and possibly, if such temperature conditions stay for a long time, decreases α-acids and formation of iso-α-acids, but the β-acids remain stable. In case of open foil, both α- and β-acids decrease as well as inert gas evaporates out of package (FORSTER, 1999; 2002; 2003a; b).

For evaluation of the brewing value of hop and α-acids stability of hop varieties, the Hop Storage Index (HSI) was developed (STEVENS, 1987). HSI indicates the hop freshness and gives an insight into hop handling until delivery (picking, drying, storage, processing and transportation). Time, temperature and hop damage have significant impact on the HSI value (FORSTER, 2001a; b; 2002; 2003a; b). Varieties of hops differ in their original ratios, but an HSI value of less than 0.28 and up to 0.31 will normally indicate that hop is fresh and has been handled well. Aged (old) hops can have an HSI index of up to 0.690 and still effect flavour characteristics of beer (FORSTER, 2001a; b). On the basis of previous consideration, it is clear that hop has to be stored in cold storage (temperature interval 0–4 °C) to maintain α-acids degradation process at a minimal level. In six months storage 4 °C, relative loss of α-acids content in hop pellets was only 8% (FORSTER, 2001b). The main goals of this study were to compare two different procedures of hop pelletisation based on the losses of α-acids and to define the effect of different storage conditions (temperature and presence of air) on losses of α-acids.

1. Materials and methods

1.1. Hop cultivar

Experiments were carried out with the dual purpose hop cultivar Aurora with balanced bitter and aromatic substances (NARZISS, 1992; FORSTER & SCHMIDT, 1994; SREČEC et al., 2001; 2004).

1.2. Hop processing and sampling

Processing of hop cones into hop pellets were carried out in two procedures: a) the usual (standard) way, which include temporary storage of dried and conditioned hop cones pressed into hop bales before pelletisation and b) the integrated system of hop pelletisation (without temporary storage of hop cones). In both cases, drying (average air temperature of 64 °C) and pelletisation of hop cones were done in the same conditions. Sampling of hop cones and hop pellets was carried out in each phase of quality chain for both procedures. It means that hop cones were sampled from the same lot after picking, kilning and pelletisation and before packing into 3-ply Al-foil bags under inert nitrogen atmosphere. Of course, during standard procedure, samples were also taken after conditioning of cones before pressing into bales and temporary storage (up to 20 °C) prior to pelletisation (usually two months). Obviously, collected samples were statistically treated as dependent samples and the link was the same growing year of hop, same cultivar, same lot with the exception of different treatments through the two different ways of pelletisation, usual and integrated.

1.3. Storage and sampling of hop pellets

In order to define the effect of different storage conditions on possible losses of α-acids, the packages of hop pellets (produced by integrated procedure) were kept in three different storage conditions. The first control samples of hop pellets, in closed packages under inert gas (N_2), were kept in brewery hop storage at 4 to 7 °C. The samples were opened gradually, one by one during one year of storage. The second control samples of hop pellets, in open bags, exposed to the atmospheric air, were kept in laboratory at room temperature (21 °C), also during one year. The third control samples also in open bags were stored in refrigerator at 4 to 7 °C during six months. During storage, all samples were analysed every month for α-acids content and HSI value.

1.4. Chemical analyses of hop cones and hop pellets

Analyses of α-acids content in hop cones and hop pellets were done by EBC Analytica-procedure 7.4 (FORSTER, 1987; 1993; ANON., 1998) and Hop Storage Index according to ASBC H-6,12 method (NICKERSON & LIKENS, 1979; ANON., 1992), respectively. All analyses were done in triplicate.

1.5. Calculation of the total losses of α-acids and statistical analyses

Total losses of α-acids content (T_A) during the hop pellets production was calculated by the following equation:

$$T_A = \frac{C_{HC} - C_{HP}}{C_{HC}} \; 100 \qquad (1)$$

where C_{HC}: content of α-acids in fresh hop cones and C_{HP}: content of α-acids in hop pellets. Differences in α-acids content during two different procedures of pellets

production (usual and integrated) were defined by *t*-test for dependent samples. Correlations between the storage time (as independent variable) and the α-acids content (as dependent variable) were defined by using the simple correlations for small samples (where x and y values were treated as $x=x_i-\bar{x}$ and $y=y_i-\bar{y}$). The reliability of analytical data was checked by the simple descriptive statistical methods (VASILJ, 2000).

2. Results and discussion

2.1. Changes of α-acids content during hop pellets production

Comparison between two different procedures of hop pellets production (usual and integrated) was done (Tables 1 and 2). Changes of α-acids content during different phases of the usual (standard) procedure of pellets production, presented in Table 1, have shown only 0.26% reduction of α-acids content during hop cones drying at average air temperature of 64 °C. Observed relatively small decrease of α-acids content was in agreement with literature data (ROSSBAUER & MÜNSTERER, 2003). After the two months of hop bales storage at temperatures up to 20 °C, significant decrease of α-acids (1.47%) content was observed as a consequence of intensive α-acids oxidation process (Table 2). During pelletisation of stored hop cones, the loss has increased by additional 0.77%. Total α-acids content has decreased from the initial 10.36% in fresh cones to the 7.86% in produced pellets (total loss 2.5%). In short, usual pellet processing resulted in 24% loss expressed in relation to total α-acids concentration found in fresh hop cones. This finding is in agreement with literature data (FORSTER, 2001b; 2002; 2003a; b) and confirms the well known fact that storage of hop cones at temperatures ≥20 °C causes significant decrease of α-acids, i.e. the higher storage temperatures the higher losses.

Table 1. Content of α-acids in hop cones and hop pellets during the phases of hop harvesting and processing into pellets type 90 by two procedures

Standard procedure	Content of α-acids (% d.w.)				
	Picked fresh hop cones	Dried hop cones	Hop cones after 2 months storage in hop bales	Hop pellets	T_A (%)
Mean ($\bar{x}$)	10.36	10.10	8.63	7.86	24
Standard deviation (s)	0.208	0.265	0.513	0.451	–
Standard error ($S_{\bar{x}}$)	0.120	0.153	0.296	0.260	–
Integrated procedure	Picked fresh hop cones	Dried hop cones	–	Hop pellets	T_A (%)
Mean ($\bar{x}$)	10.64	10.36	–	9.90	7
Standard deviation (s)	0.237	0.288	–	0.163	–
Standard error ($S_{\bar{x}}$)	0.089	0.109	–	0.062	–

Table 2. Comparison of analytical data in different phases of standard and integrated hop pelletisation procedures

Standard procedure/ Statistical parameter	Dried hop cones vs. hop cones after 2 months storage in hop bales	Hop cones after 2 months storage vs. hop pellets	Picked fresh hop cones vs. hop pellets
Difference (D)	1.47	0.77	2.50
Standard difference (*Sd)*	0.305	0.153	0.346
Probability (p) (Significance P<0.05)	0.014	0.013	0.0063
Integrated procedure/ Statistical parameter	Picked fresh hop cones vs. dried hop cones	Dried hop cones vs. hop pellets	Picked fresh hop cones vs. hop pellets
Difference (D)	0.28 (ns)	0.46	0.74
Standard difference (*Sd*)	0.393	0.269	0.321
Probability (p) (Significance P<0.05)	0.103	0.004	0.0008

ns: not significant

Changes of α-acids content during the integrated procedure of hop pellets production, also presented in Table 1, confirm that integrated procedure considerably decrease loss of α-acids in comparison to the usual procedure. This is a consequence of hop cones pelletisation immediately after drying, and there was no time for α-acids oxidation. Total loss of α-acids during integrated procedure of hop pellets production was only 0.74% (Table 2) or α-acids concentration has decreased by 7% compared to the initial concentration in fresh cones. Obviously, integrated procedure is more appropriate for hop pellets production than the usual one. Significant reduction of α-acids losses is also important from economical point of view, as the price of hop pellets depend on their α-acids content. Furthermore, in respect to the statistical parameters of data variability (FORSTER, 2001a), the share of α-acids was more homogeneous in pellets produced by integrated than by the standard procedure.

2.2. Changes of α-acids content during hop pellets storage in different conditions

As it can be seen in Fig. 1, the α-acids content of hop pellets stored in closed packages under inert gas (N_2) at 4–7 °C have fallen only 0.2% (or 2% expressed on total value) during the storage period of one year. This is not statistically significant ($t_{0.05}$=0.576). The value of HSI for this hop pellet sample was 0.33 (Table 3). Obtained results are in agreement with literature data (KALTNER et al., 2001; IKEDA et al., 2002; FORSTER, 2003a; b). The second group of the samples exposed to the air in open packages at 21 °C (Fig. 1) has shown significant decrease of α-acids. During one year of storage, α-acids content decreased by 54% compared to the initial α-acids content and the value of HSI increased up to 0.59 (Table 3). Therefore, this sample can be classified as "very old hop pellets" (FORSTER, 2001b). The third group of the samples stored in open packages at 4–7 °C has also expressed decrease of α-acids content (Fig. 1), but it was considerably lower than at 21 °C. During six months of storage, total loss of α-acids was only 10.5% expressed on initial total α-acids content in pellets. The HSI value was

0.35 (Table 3) and therefore pellets are classified as "slightly aged hop pellets" (FORSTER, 2001b). Results clearly confirmed influence of temperature and air on α-acids degradation during hop pellets storage, as have already been found by other studies (FORSTER, 2001a; b; 2002; 2003a; b).

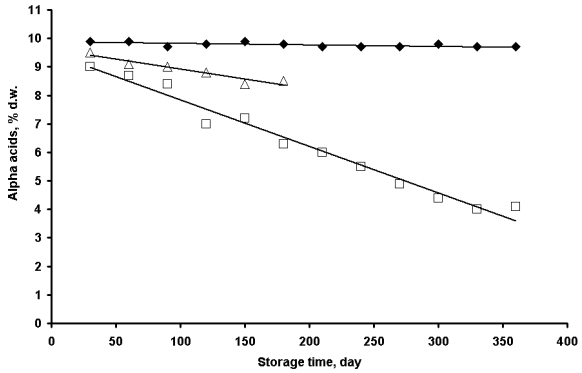

Fig. 1. Correlation between losses of α-acids and storage time of hop pellets kept under different conditions.
♦: Closed packages (N_2 atmosphere) at 4–7 °C (r=–0.49 ns; $t_{0.05}$=0.576; ns: not significant);
□: open packages (air presence) at 21 °C (r=–0.98; $t_{0.01}$=0.708);
Δ: open packages (air presence) at 4–7 °C (r=–0.93; $t_{0.05}$=0.811; $t_{0.01}$=0.917).
Solid lines represent correlation between experimental data

Table 3. Comparison of HSI values in hop pellets stored at different conditions

Statistical parameter	HSI		
	Closed packages (N_2 atmosphere) at 4–7 °C during 365 days	Open packages (air presence) at 21 °C during 365 days	Open packages (air presence) at 4–7 °C during 180 days
Mean ($\bar{x}$)	0.33	0.59	0.35
Correlation coefficient *(r)*	0.47 (ns)	1**	0.50 (ns)
N=4			
(N-2=2)			
$t_{0.05}$	0.950	0.950	0.950
$t_{0.01}$	0.990	0.990	0.990

ns: not significant; *P=0.05; **P=0.01.

3. Conclusions

Integrated procedure of hop pellets production is more efficient than the usual (standard) one due to the significant reduction of α-acids losses. Furthermore, the distribution of α-acids content in pellets produced by integrated process was more

homogeneous than by the usual one. The highest decrease of α-acids content from pellets was observed during storage at 21 °C in the presence of air and the lowest one in tightly closed samples under inert gas (N_2) at 4–7 °C, respectively. The most important parameters for α-acids degradation are temperature and air (oxygen) presence.

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