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Impact of Process Parameters and Bulk Properties on Quality of Dried Hops

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Abstract: Hops are critical to the brewing industry. In commercial hop drying, a large bulk of hops is dried in multistage kilns for several hours. This affects the drying behavior and alters the amount and chemical composition of the hop oils. To understand these changes, hops of the var. Hallertauer Tradition were dried in bulks of 15, 25 and 35 kg/m² at 60 °C and 0.35 m/s. Additionally, bulks of 25 kg/m² were also dried at 65 °C and 0.45 m/s to assess the effect of change in temperature and velocity, respectively. The results obtained show that bulk weights significantly influence the drying behavior. Classification based on the cone size reveals 45.4% medium cones, 41.2% small cones and 8.6% large cones. The highest ΔE value of 6.3 and specific energy consumption (113476 kJ/kg$_{H2O}$) were observed for the 15 kg/m² bulk. Increasing the temperature from 60 °C to 65 °C increased the oil yield losses by about 7% and myrcene losses by 22%. The results obtained show that it is important to define and consider optimum bulk and process parameters, to optimize the hop drying process to improve the process efficiency as well the product quality.

Keywords: bulk weight; hop cones size distribution; chemical analysis; energy consumption

1. Introduction

Hop (Humulus lupulus) is a perennial climbing plant that is used in the brewing industry to impart a bitter taste and provide a "hoppy" aroma to beer [1]. About 97% of the world's hop crop is used for beer making [2]. Initially, hops were primarily used to provide stability to beer and increase shelf life. However, with extensive research and knowledge acquired over time, hops have also increasingly been used for other purposes such as creation of foam characteristics [3]. The bittering acids, tannins, chemical and aroma compounds are situated in the lupulin glands of the hop cone [1,4]. As of 2016, hops can be spilt into three categories namely aroma hops, bitter hops, and dual-purpose hops. Aroma hops are responsible for contributing fruity, piney, citrusy, and tropical aromas to the beer while bittering hops are associated with its bitterness [5,6]. Dual-purpose hops are described as bitter hops with special aroma characteristics [6]. However, freshly harvested hop cones have a high water content, which in turn promotes microbial spoilage and thus, cannot be stored for an extended period of time [7]. Therefore, to ensure minimal post-harvest losses and consistent

product quality, hop cones need to be processed immediately after harvesting. In the Hallertau region, 22 hop varieties are grown [6]. As harvest lasts for a maximum of eight weeks a year, and with harvesting period for each variety lasting for less than two weeks, the efficient processing of harvested hops is of great economic importance to growers [8].

To extend shelf life and increase stability of hops, convection drying is used as a preservation technique by hop growers. Hop drying is a complicated process due to both the physical and chemical characteristics of the hop cone. The anatomy of the hop cone consists of strig as its main axis, followed by bracteole which is made up of sheets of fine petals adhering and covering the strig and contain the lupulin glands and finally the bract as the outer structure of the hop cone which consists the hop leaves. Compared to the mass, the bracteole and bract account for relatively larger surface area of the cone. The strig, however has an extremely small surface area. During the drying process, the strig is shielded from the drying air due to the exterior bract and thus leading to high-water content retention. Furthermore, the cone size and shape, strig shape, arrangement of the cone leaves and density of the capillaries in the bracteole differ in individual hop varieties and thus further complicate the drying process [9]. According to [9], at the end of the drying process if a hop cone has a water content of 8–9%, the water content within the bracteole is 4–7%, while that within the strig is up to 20–30%. The larger amount of water within the strig is released to the bracteole and bracts after the drying process during ventilation in the conditioning chamber [9]. This stage allows for the establishment of constant moisture equilibrium between the strig, the bracteole and bract [10]. Therefore, the stage of conditioning is of utmost important in commercial drying of hop cones. Optimum process parameters during hop drying are of paramount importance as over drying of hops (moisture content ≤ 7%) can increase lupulin losses due to the shattering of hop cones [11]. Conversely, stopping the drying process at moisture contents ≥ 13% would initiate microbial spoilage. Furthermore, the temperature of the drying process also influences quality parameters such as color, oil yield and oil composition of the produce.

Traditionally, hops were dried in a single layer (3–5 cm). The drying time required was largely dependent on the weather conditions and usually ranged between two to ten days [7]. With improvement in technology and understanding of the main characteristics for hop drying, this traditional method was replaced by multistage kiln and belt driers. In belt driers, the hops are dried in three layers in a continuous process. The fresh hops are fed onto the uppermost belt, while the dried hops remain on the lowest belt until the final moisture content is achieved. For optimum drying using belt driers, it is important to evenly feed fresh hops over the entire belt width. Unlike the continuous process of belt driers, multistage kiln drying is a batch process [10]. The drier consists of three to four superimposed perforated trays, through which hot air is passed (bottom to top) to reduce the moisture content within hops. Freshly harvested hop cones are filled within the topmost tray and as the drying progresses, the bulk of hop cones is transferred to the trays below. Once the drying process is ended, the dried hop cones are transferred into a conditioning chamber. Depending on the variety in focus and the drying system, the drying time with multi stage kilns can vary between five to eight hours [7]. One of the biggest problems with kiln driers is potential uneven drying of the hop cones due to the formation of so called "nests". Nest generation is the formation of compacted zones within the hop stack and can lead to varying air resistances from the beginning of the drying process. Parameters such as air velocity, bulk height and density as well as air temperature are the influencing process parameters that need to be considered and optimized to avoid the formation of nests [9].

With hops being so important to the brewing industry, the need for optimisation of the drying process and understanding of the changes within the product during drying is imminent. Currently, to determine moisture content, temperature and humidity sensors are placed within the hop stack. However, this technique lacks in providing information of the overall stack and thus, leads to not detecting potential variations of moisture content within the hop stack [10]. With advancement in technology, smart drying systems that combine non-invasive measurement techniques with process control have also been developed. Implementation of non–invasive techniques such as computer-aided vision (CAV), thermography (TI), laser backscattering (LB), and hyperspectral imaging (HSI)

have demonstrated the ability to analyse the physical and chemical properties, colour, shrinkage, texture, shape, water content, porosity, defects and firmness of the product [12]. A recent investigation performed also used color and HSI imaging as non-invasive techniques to monitor quality parameters such as color of hops of different bulk weights, provide spectral correlations and build models for prediction of selected chemical components. This study has shown promising results for novel real time monitoring during the drying of hops [3]. Applications of thermal imaging in kilns have also been used to understand the drying behavior of the hop cones [9,13] and further potentially combine them with control systems for real time product based control of the drying process.

Drying is an energy intensive process and in case of hop drying, fresh air is heated to 63 °C–68 °C using fuel oil or gas burners [7]. On average, 44 litres of fuel oil is currently required per 100 kg of dried hops. With increasing fuel prices, alternative energy sources and heat recovery systems are becoming of larger interest [14]. In the context of hop drying, options such as preheating of air, use of waste heat from power generator, use of combined solar-building heat and heat recovery from exhaust air are some of the considered alternatives. However, the economic efficiency of these alternative energy sources largely depends on the acquisition costs, operating costs, and equipment life expectancy [9]. Another option to reducing heating oil consumption was investigated by performing experiments with inclusion of a heat recovery system and optimisation of drying process parameters. The results for the performed experiments reveal a potential of 20% savings in heating oil [7,9]. The results from these experiments successfully led to the installation of heat recovery systems on industrial hop kilns and further led to the development of a pilot plant scale dryer that considers better control of the process parameters and gentler kilning of the hop cones [7].

The experiments conducted within this study were performed on the aforementioned pilot plant dryer and aimed to understand the effect of varying bulk weights and hop size distributions on the drying behavior of hops and the associated changes in quality parameters such as color, hop oil yield and hop oil content. As energy is a crucial aspect of the drying process, specific energy requirement for the different bulk weights were also calculated.

2. Materials and Methods

Investigations were performed at the Bavarian State Research Center for Agriculture (LfL), Hop Research Centre Hüll, Wolnzach. Organic aroma hops of the variety Hallertauer Tradition were used for investigation. Based on the harvest dates for this variety in 2018, the experiments were conducted for a week starting from end of August to beginning of September. Hop vines were freshly harvested prior to each investigation and further mechanically de-vined using a hop cone harvester (Hopfenpflückmaschine Wolf WWE 220, Giesenfeld, Germany).

2.1. Dryer

The pilot plant scale dryer described by [3,7] was used for conducting the experiments within this study (Figure 1). The dryer consisted of two drying layers and a slip case which is also considered as a drying stage. However, in case of this experimental investigation, the second drying layer was not used, and the slip case was only used to remove the hop cones. Thus, the overall drying process was conducted using the top drying layer itself. This means that the hop cones were not tipped into the second drying layer but were dried continuously on the top layer and tipped directly into the slip case at the end of drying. Temperature and humidity of the air entering and exiting the top drying layer was measured using Testo 174 H data loggers with an accuracy of ±0.5 °C for temperature and ±3% for relative humidity (Testo SE & Co. KGaA, Lenzkirch, Germany).
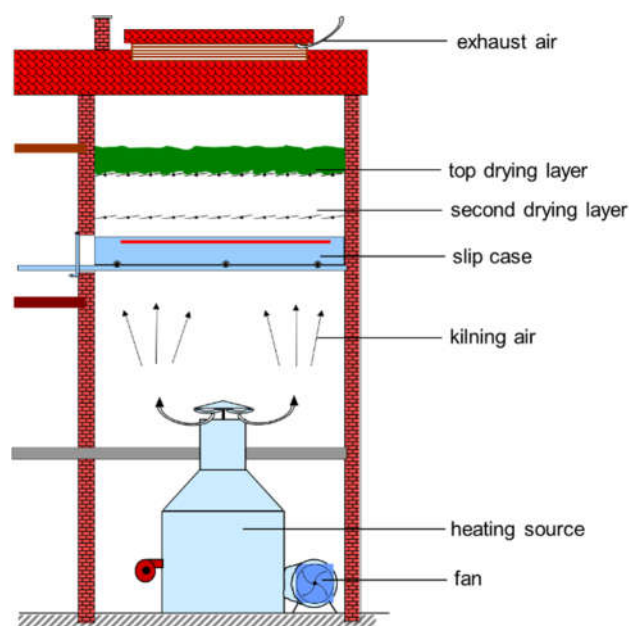
Figure 1. Schematic of hops dryer (adapted and revised from [9]).

Experimental investigations were conducted for hops with bulk weights of 15, 25 and 35 kg/m² at 60 °C and 0.35 m/s, respectively. The 25 kg/m² bulk was considered as the control bulk and changes to the velocity to 0.45 m/s and temperature of 65 °C were only performed at this bulk weight. Thus, allowing for better comparison for further analysis. The drying time for each bulk weight was estimated based on the set final moisture content of $\cong$ 7–8% (equivalent moisture ratio [MR] $\cong$ 0.10–0.12). For reproducibility and repeatability, three repetitions were performed for all experiments. Table 1 provides a summary of the experimental plan for the investigations conducted.

Table 1. Experimental plan for drying of Hallertauer Tradition.

Temperature [°C]-Velocity [m/s]	Bulk Weight [kg/m²]		
	15	25	35
60-0.35	X	X	X
60-0.45		X	
65-0.35		X	

2.2. Sampling

For further analysis, the samples were extracted from the front section of the dryer at defined time intervals. At each time interval, a small set of approximately 50 g was extracted from the dryer. To dissuade the formation of artificial holes due to sample extraction, the hop cones were redistributed throughout the bulk. The initial moisture content of hops for the different bulk weight varied between 78% and 80%. For moisture content analysis, the samples were collected at a 20 min interval from the start for the first hour, followed by a 30 min interval for the next hour, and a 60 min interval thereafter. End time for the drying process varied depending on the weight of the bulk. Table 2 provides details of the sampling intervals for the different experiments.

Table 2. Sampling intervals for different experiments.

Interval	60 °C-0.35 m/s			60 °C-0.45 m/s	65 °C-0.35 m/s
	15 kg	25 kg	35 kg	25 kg	25 kg
	Time Section in Minutes				
20 min	0–60	0–60	0–60	0–60	0–60
30 min	60–120	60–120	60–120	60–120	60–120
60 min	120–180	120–180	120–180	120–180	120–180
End	180	210	240	200	180

The samples extracted at different intervals were further placed in an oven at 105 °C for 24 h in accordance with the official Association of Official Analytical Collaboration (AOAC) method [15] to obtain the final moisture content of the samples. The moisture content on dry basis (MC_{db}) was calculated using Equation (1).

$$MC_{db} = \frac{m_t}{m_{dm}} - 1 \tag{1}$$

where m_t is the total mass of a sample at a given time and m_{dm} is the absolute dry matter in a sample. In addition to MC_{db}, the moisture ratio (MR) of the samples was also calculated as it allows to compare the different samples with each other by normalizing the moisture contents to their initial moisture contents and thus displaying them in values ranging from one to zero. MR was calculated using Equation (2).

$$MR = \frac{MC_{db,1} - MC_e}{MC_{db,0} - MC_e} \tag{2}$$

where $MC_{db,1}$ is the dry basis MC at a given time, $MC_{db,0}$ the dry basis MC before drying and MC_e is the equilibrium moisture content. As the influence of MC_e is relatively small (≤2%) [16], the MR equation is further simplified to Equation (3).

$$MR = \frac{MC_{db,1}}{MC_{db,0}} \tag{3}$$

Sampling intervals similar to those described for moisture content analysis were used for color acquisition. A detailed explanation on the color measurements is provided in Section 2.3.

Hop cone size varies both between and within the varieties. This variation is evidently observed from Figure 2 wherein hop cone sizes for two varieties namely Hallertauer Tradition and Hallertauer Herkules is shown.

(**a**) (**b**)

Figure 2. Hop cone size variation in varieties (**a**) Hallertauer Tradition, (**b**) Hallertauer Herkules [9].

Therefore, to understand the distribution of the hop cone size, 50 g of fresh hops were extracted from the bulk and sorted based on their length and diameter. Cones smaller than 2 cm in length were classified as small, between 2 cm–3.2 cm as medium and larger than 3.4 cm as large. The classified cones were further weighed to establish their ratios within the 50 g mix. The investigation of hop cone size distribution was limited to fresh hop cones (i.e., 0 min) as the aim of this study was to investigate the effect of hop cone size on the drying process.

For distillation of essential oils, samples were collected prior to drying (fresh) and at the end (dried) of each trial. Distillation of essential oils was carried out only for the control bulk i.e., 25 kg/m² bulk dried at air velocity of 0.35 m/s and at 60 °C and 65 °C, respectively, to assess the effect of temperature on the overall hop oil yield and hop oil composition. Finally, to mimic the commercial drying process, it was ensured that the bulk was thoroughly mixed using a rake prior to sample collection.

2.3. Imaging

During the drying process, hop cones undergo significant color change. To understand the development of color, hop samples were placed in a photo box (Life of Photo 60 cm LED Light Cube, Weiwa Foto, Gummersbach, Germany). The photo box includes an array of 160 LED lamps as the illumination and a window for the camera. To capture the images, an RGB Camera (model no. 61BUC02, The Imaging Source Europe GmbH, Bremen, Germany) was placed at a distance of 29 cm from the surface of the product [17] using the camera window of the photo box. A schematic of the imaging system used is presented in Figure 3.
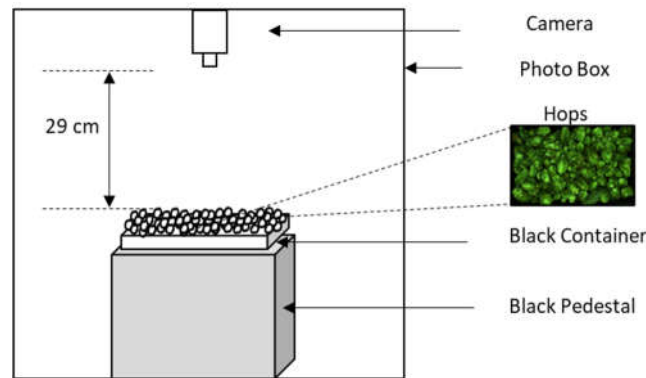
Figure 3. Schematic of RGB camera imaging system for hops.

The acquisition of the images was realized using the IC Capture software (the Imaging Source Europe GmbH, Bremen, Germany). The captured images were further processed in MATLAB® (The MathWorks, Inc., Natick, MA, USA) to convert RGB values to CIE L*a*b* values. For this purpose, RGB images were converted to gray and further into binary images (0, 1) using appropriate threshold factors. In the binary images, the values 0 and 1 were assigned to the background and the sample, respectively. To remove any undesirable objects, the function bwareaopen was applied. The images were then masked to ensure that all values now belong to the image and no values are being assigned to the background. Finally, the function rgb2lab was used to convert the RGB values into CIE L*a*b* values for the images captured [18]. The function rgb2lab within MATLAB® uses a two-step process. In the first step, RGB values are converted into XYZ space using linear matrix in Equation (4) [19].

$$\begin{bmatrix} X \\ Y \\ Z \end{bmatrix} = \begin{bmatrix} 0.412453 & 0.357580 & 0.180423 \\ 0.212671 & 0.019334 & 0.072169 \\ 0.019334 & 0.119193 & 0.950227 \end{bmatrix} \begin{bmatrix} R \\ G \\ B \end{bmatrix} \tag{4}$$

The XYZ values thus obtained are further normalized to the D65 white points. In the final step, the XYZ values are converted to CIE L*a*b* values using Equations (5)–(7) [20].

$$L^* = 116 \times \left(\frac{Y}{Y_n}\right)^{\frac{1}{3}} - 16 \quad \text{if } \left(\frac{Y}{Y_n}\right) > 0.008856 \tag{5}$$

$$116 \times \left(\frac{Y}{Y_n}\right) \qquad \text{if } \left(\frac{Y}{Y_n}\right) > 0.008856$$

$$a^* = 500 \times \left[\left(\frac{X}{X_n}\right)^{\frac{1}{3}} - \left(\frac{Y}{Y_n}\right)^{\frac{1}{3}}\right] \tag{6}$$

$$a^* = 200 \times \left[\left(\frac{Y}{Y_n}\right)^{\frac{1}{3}} - \left(\frac{Z}{Z_n}\right)^{\frac{1}{3}}\right] \tag{7}$$

where L*, a* and b* represents lightness, red/green and yellow/blue, respectively. Xn, Yn, and Zn are the values of the blank references.

To compare the non-invasive method to a gold standard color measurement method, a CR-400 Chromameter (Konica Minolta Sensing Europe B.V., München, Germany) in combination with the SpectraMagic NX Software (Konica Minolta Sensing Europe B.V., München, Germany) was also used. For calibration, a ColourChecker Classic chart (X-rite Gmbh, Planegg-Martinsried, Germany) was used to compare the values obtained from the chromameter and the RGB camera for the 24 color patches.

Finally, the values obtained from each of the repetitions were further averaged to calculate the total color difference (ΔE_{Lab}) using Equations (8)–(11):

$$\Delta E_{Lab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \tag{8}$$

$$\Delta L^* = L_1^* - L_0^* \tag{9}$$

$$\Delta a^* = a_1^* - a_0^* \tag{10}$$

$$\Delta b^* = b_1^* - b_0^* \tag{11}$$

where L^*_1, a^*_1, b^*_1 is the lightness, red/green and blue/yellow at the specific time interval and L^*_0, a^*_0, b^*_0 is the initial lightness, red/green and blue/yellow [21].

2. 4. Gas Chromatography

Gas Chromatography (GC) analysis was performed using a Dani GC with flame ionisation detection (FID; DANI Instruments SpA, Milan, Italy) with a defined temperature program. The gas flow was divided in a ratio of 1:25 (spilt) with helium being used as the carrier gas. The injector and detector temperature were set to 200 °C. The GC unit was also calibrated using the internal standard developed by the Hops Research Center, Wolnzach. Myrcene, linalool, ß-caryophyllene and humulene—representing the most valuable oils related to Hallertauer Tradition—were the main components analysed in the GC unit. Mathematical calculation for quantity of hops required for distillation of essential oils was determined using the method described in [17].

2. 5. Energy Consumption

To calculate the specific energy consumption per bulk weight it was essential to first calculate the amount of dry matter at the end of the drying time. To this end, Equations (12) and (13) were used to calculate the dry matter in the bulk:

$$DM_i = \frac{100 - MC_f}{100} \tag{12}$$

$$DM_f = DM_i \times W_i \tag{13}$$

where DM_i is the amount of dry matter per kg, MC_f is the final moisture content, DM_f is the amount of dry matter in the bulk (kg) and W_i is the initial weight of bulk.

The specific energy requirement per bulk weight was calculated using Equations (14) and (15) [22,23]:

$$E_t = A \times \vartheta \times \Delta T \times \rho_a \times C_{p,a} \times t \tag{14}$$

$$E_s = \frac{E_t}{W_i - DM_f} \tag{15}$$

where E_t is the total energy consumed (kJ), A is the area of the drying chamber, v is the velocity of the air entering the drying chamber (m/s), ΔT is the temperature difference between the inlet air and surrounding air (K), ρ_a is the density of air (kg/m³), $C_{p,a}$ is the specific heat capacity of air (kJ/kg K), t is the run time (s), E_s is the specific energy required (kJ/kg) and W_i is the initial weight of the bulk.

3. Results and Discussion

3.1. Drying Behavior

Figure 4 presents the temperature and the humidity profile of the air exiting the drying chamber. From the extracted results, it is observed that the humidity for all bulks other than that for 15 kg/m^2 is close to 100% at the initial phase of drying. As the drying continues, the temperature of the air exiting (top layer of the hop bulk) begins to increase and the humidity to decrease. The time required for the temperature to reach the set drying temperature significantly depends on the temperature as well as the bulk weight. The lower the temperature and the higher the bulk weight the longer the time required for drying and vice versa.
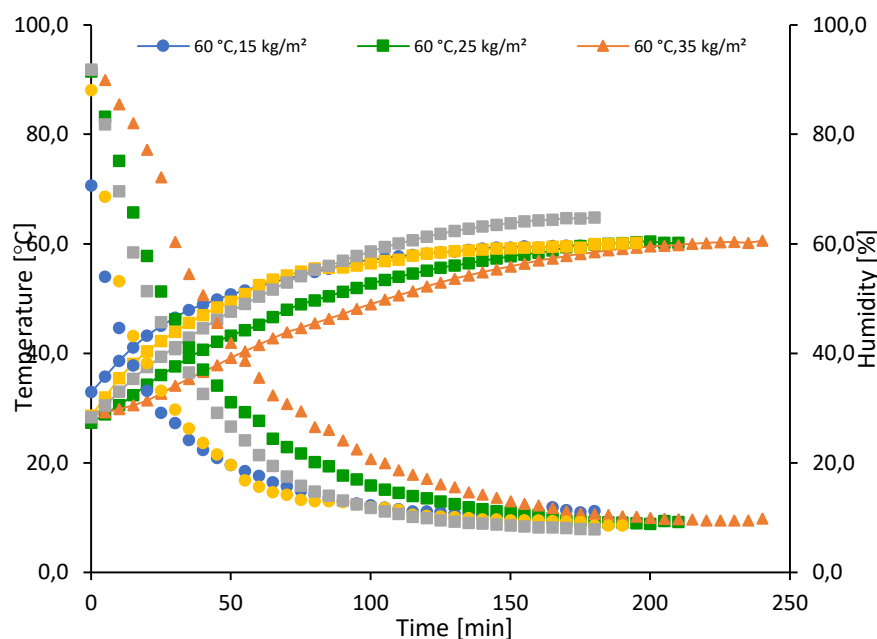
Figure 4. Temperature (symbol and line) and humidity (symbol) profile for different bulk weight and different process settings.

Figure 5 presents the drying curves for different bulk weights of hops dried at 60 °C and 0.35 m/s air velocity. The bulk weight had a significant influence on the drying time required to attain the set moisture ratio of 0.12. Hops of 15 kg/m^2 bulk weight required 180 min while 25 kg/m^2 and 35 kg/m^2 bulks required 210 min and 240 min, respectively. Similar results were also obtained by [3] for hops with bulk weights of 12 kg, 20 kg and 40 kg dried at 60 °C and 0.35 m/s. Overall, the development of the drying curve for all three bulks is characteristic to the curves obtained from convective drying process; with rapid losses in moisture content during the initial phase of drying and eventual reduction as it transitions from the first to the second phase of drying. In the case of hops, this development of drying behavior can be associated to two factors: structure of hop cone and the variety in focus. As mentioned previously, the structure of the hop cone plays a crucial role in the drying behavior. The strig which contains high water content and is surrounded by the bracteole and bract of the hop cone, only begins to dry once the bracteole (leaves of the hop cone) has reached a specific moisture content. It is believed that the development of high suction tension acts as the driving force to draw the moisture from the strig [3] and thus, resulting in specific drying behavior. Furthermore, the weight percentage ratio between the strig and the bracteole also depends on the variety in focus. In case of Hallertauer Tradition, this ratio varies from 9–10% [24]. In addition to the variety, harvesting conditions such as environmental temperature, humidity, location of harvest, soil quality, hop cone size and shape also affect the overall hop cone drying behavior [3]. The effect of hop size, shape and distribution on the overall drying behavior has been further explained in Section 3.2.
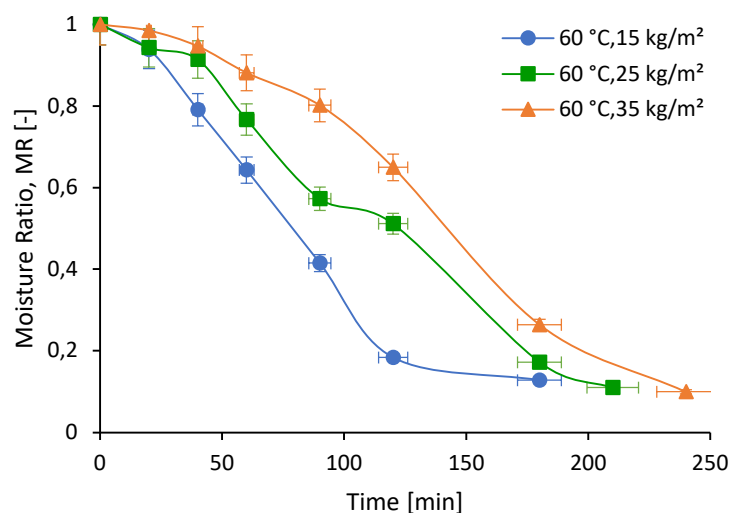
Figure 5. Moisture ratio (MR) vs. time (min) for hops with three different bulk weights dried at 60 °C and 0.35 m/s.

Figure 6 presents the results obtained from changing the velocity (Figure 6a) and changing the temperature (Figure 6b) for 25 kg/m² bulk of hops.
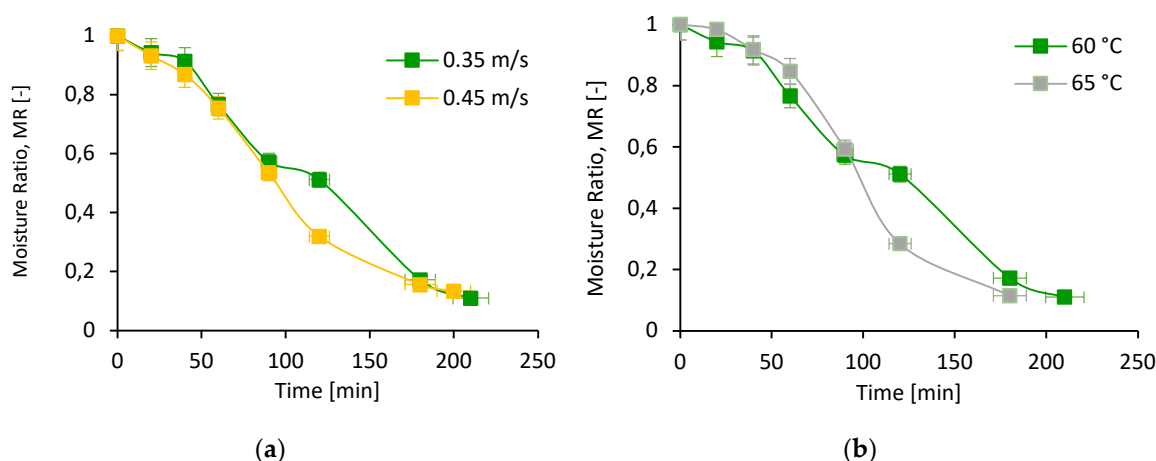

(**a**)

(**b**)

Figure 6. Moisture ratio (MR) vs. time (min) for hops dried at (**a**) 0.35 m/s and 0.45 m/s at 60 °C (**b**) 60 °C and 65 °C at 0.35 m/s.

No significant impact on the drying time between air velocity of 0.35 m/s (210 min) and 0.45 m/s (200 min) was observed. This is contradictory to several investigations performed through the years that identify air velocity to be an influencing factor for reducing the drying time [20,25]. For most of these studies, a thin layer convective drying of products is investigated as compared to a bulk of 25 kg/m². The insignificant influence of air velocity on the bulk could be due to the air being completely saturated with water vapor prior to reaching the top of the bulk. Thus, the deficit in saturation of the drying air acts as the limiting factor as compared to the air velocity [26]. This further explains the similarity in drying behavior for the first 90 min and then the observed rapid moisture losses for 0.45 m/s for the rest of the drying period. However, increasing the air velocity is also not an ideal solution to reduce the drying time as increasing the velocity leads to faster drying at the surface as compared to the center, in turn stiffening the surface and causing uneven shrinkage of the product [27]. Furthermore, increasing velocity also increases the overall energy demand for the drying process [9].

Changing the drying temperature, the overall drying time is observed to have reduced from 210 min for 60 °C to 180 min for 65 °C. This is analogous to the several investigations performed on the influence of drying temperature on the drying time [20,23,28–30]. From the drying curve presented

in Figure 6b, it is also observed that the moisture losses were rapid for 60 °C than 65 °C for the initial phase (up to 60 min), indicating that the 60 °C bulk dried faster than the 65 °C bulk. Within the 7-day investigation period, a severe thunderstorm on Day 3 led to harvesting of wet hops, thus increasing the overall moisture content of the hop cones. Furthermore, as the hop cones were harvested just prior to the beginning of each drying trial, it was observed that those harvested in the morning had a higher moisture content due to the morning dew as compared to those harvested after midday. The trial period within the experiments were randomized to ensure that each repetition was performed on a different day and at a different time. However, the difference in the environmental conditions and time for 65 °C trials could have led to the observed results. A study performed on the impact of environmental conditions on the hop cones during drying, shows that weather conditions such as heavy rains, morning dew and sunny days significantly influence the drying process and the overall product quality [3].

3.2. Hop Cone Size

Results from the classification of average hop cone sizes within a hop stack is presented in Table 3.

Table 3. Average hop cone size distribution within a hop stack.

Cone Size	Average Cone Length [cm]	Average Cone Diameter [cm]	Average Weight of One Cone [g]	Average Hop Weight [g]
Small	1.6	0.9	0.3	20.6
Medium	2.7	1.3	0.7	22.7
Large	3.9	1.3	1.1	4.3
Total Weight				47.57

For the Hallertauer Tradition, the distribution of cone size lies between small to medium with small hop cones accounting for 41.2%, medium for 45.4% and large for 8.6% of the total measured weight. A total of 50 g of hops were weighed for classification of which 4% were lost due to the cones either being broken or shattered. In addition to the size, a linear increase in the average weight of one cone is also observed. A study conducted on drying of coarse lignite particles of varying particle size indicates that larger particle sizes require longer drying times as compare to the small particles [31]. Particles smaller in size have a larger specific surface area which results in faster heating rate and better transfer of heat from the drying medium to the center. Additionally, the moisture transfer from inside to the surface is also faster within smaller particles, while smaller diameter also provides less resistance to the internal heat transfer [32], thus, leading to faster drying processes. However, bulks with a large number of small particles such as that investigated within the current study, tend to increase resistance within the bulk due to a decrease of average particle size and an increase of void fraction, and thus the associated increase in pressure drop. Investigations performed on grain drying showed that the grain surface characteristics can potentially tend to increase the airflow resistance [33]. For locust beans at low moisture content, the cotyledons tend to become hard and smooth which in turn increases the frictional losses due to high resistance to airflow [34].

Furthermore, the degree of compaction of the hop cones also increases with bulk height. This results in a rapid decrease in the air flow rate and the drying capacity while simultaneously increasing the retention time of the hop in the top layer. As drying progresses, the cones tend to become solid and maintain their individual shape and their position in relation to each other in the top layer. This results in the creation of air channels and formation of zones with uneven air flow [9]. In the current investigation, the proportion of small size cones is significantly high and hence the degree of compaction within the stack is also high. Although no air channels formation was observed in 25 or 35 kg/m² stack, relatively many air channels were observed to have formed in the 15 kg/m² bulk. Figure 7 shows the formation of air channels within the bulk.
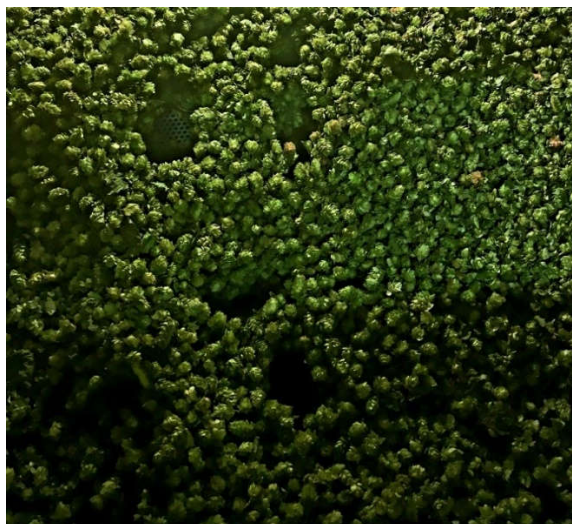
Figure 7. Air channel formation in a 15 kg/m² bulk of hops.

The overall bulk height of a 15 kg/m² is not high. Therefore, bulks with low heights such as those for 15 kg/m² combined with high numbers of small sized cones and set air flow rate can also lead to the formation of air channels. A study performed on pistachio nuts also revealed a linear increase in air flow resistance with increasing depth of the pistachio column. The study also adds that the air flow rate significantly influenced the pressure drop within the column as compared to other parameters such as moisture content, particle interaction and filling method [35]. The inclusion of a higher number of small cones resulted in faster drying but also led to the formation of air pockets or so called "nests" within the drying chamber which in turn led to uneven drying [10]. Investigations performed on storage of hops revealed that hops stored for a longer amount of time undergo oxidation which lead to formation of compounds that interfere with the aromatic smell associated with hops [17]. Bulks with unevenly distributed moisture content can also undergo a similar degradation and thus leading to significant losses of both physical and chemical attributes of the product. Therefore, in commercial hop drying units, to avoid formation of large air pockets, low bulk weights such as 15 kg/m² are not usually considered. Furthermore, in the commercial process, the air velocity is also lowered as the drying progress so as to ensure even drying of hop cones and deter the formation of air channels. Moreover, during extraction of samples at specified intervals, a number of small hop cones were observed to have exited the drying chamber. This could be due to two reasons. Firstly, as mentioned earlier, small cones have large surface area and hence dry faster. Secondly, the pressure difference within the drying chamber and the surrounding environment could have also led to the dried small hop cones to exit the dryer.

As the size of the hop cone is an uncontrolled parameter, a solution to optimize the drying performance, and reduce the air resistance, is to fill low bulks followed by tipping at shorter interval. Thus, aide in loosening the compacted layers within the bulk and reducing air resistance which increases the air flow and the drying performance [9].

3.3. Chemical Analysis

3.3.1. Oil Content

The variety Hallertauer Tradition is a product of cross breeding between the variety Hallertauer Tradition Gold and a male breeding line. It has a slightly flowery, herby-spicy aroma with an amount of oil ranging between 0.005 L/kg–0.007 L/kg [6,9]. Within the experimental investigation conducted, the amount of oil extracted for fresh hops ranged between 0.0025–0.0027 L/kg while those for dried hops ranged between 0.0023–0.0024 L/kg. Table 4 summarizes the amount of oil extracted and the standard deviations for the investigations conducted.

Table 4. Amount of oil extracted in L/kg with standard deviations (SD) for prior and after drying process.

Temperature [°C]	Amount of Oil [L/kg hops]	
	Fresh	Dried
60	0.0024	0.0023
SD	0.0006	0.0005
65	0.0027	0.0024
SD	0.0003	0.0004

As compared to the dried samples, significant deviation is observed within the fresh samples which could be due to the natural heterogeneity of hops within and between the different bulks. Figure 8 presents the amount of oil extracted prior to drying and after drying at 60 °C and 65 °C, respectively.
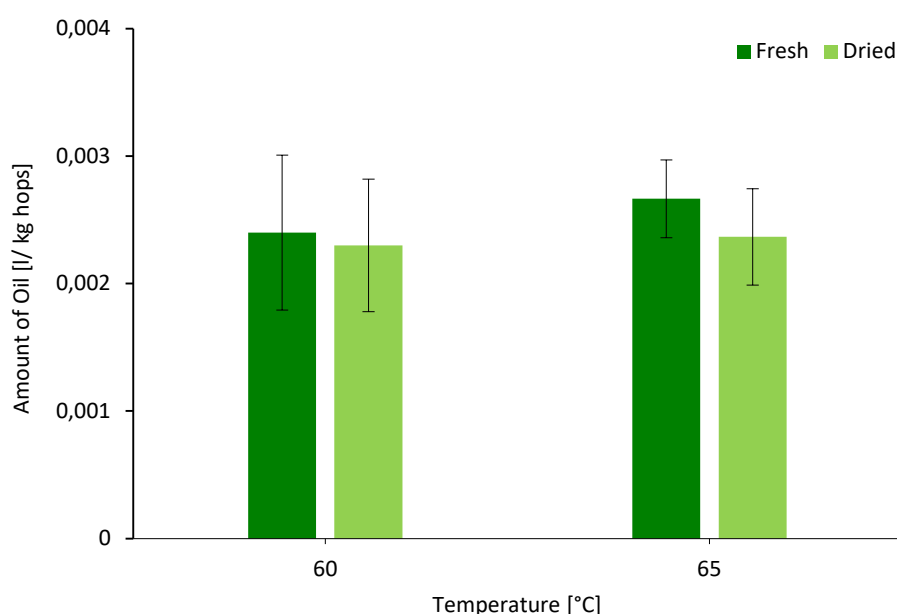
Figure 8. Amount of oil in L/kg hops extracted for samples extracted at 0 min and 210 min for hop cones dried at 60 °C and 65 °C at 0.35 m/s.

The overall amount of oil extracted for fresh hops was lower than that reported in literature (0.007 L/kg) [6]. Factors such as location, environmental conditions at the location and harvest time have been shown to have a significant effect on the total amount of oil [36,37]. In case of the Hallertauer Tradition, a five-week investigation conducted for three continuous years for essential oil content reveals that the amount of oil extracted is lower at the initial harvest time (about 0.005 L/kg) and increases over time (about 0.02 L/kg) until it reaches an optimum point (roughly Week 3). Harvesting after this period has also shown a decrease in the overall amount of oil [38]. The investigation within this study was performed in the first week of the harvesting period for Hallertauer Tradition. The early harvest combined with associated environmental conditions could have led to the lower amounts of oil extraction.

Drying temperature also had a significant effect on the overall hop oil content. Within the current investigation it was observed that increasing the drying temperature decreased the total amount of oil extracted after drying. An investigation performed on the Mandarina Bavaria variety of hops revealed losses in the total oil extracted after drying as compared to prior to drying [17]. Furthermore, drying of Laurel (*Laurus nobilis* L.) leaves at either 45 °C or 65 °C accounted for losses close to 83% [39]. An experimental study performed on drying the aerial parts of sweet wormwood (*Artemisia annua* L.) also revealed   a decrease in the yield of oil with an increase in drying temperature [40].

3.3.2. Gas Chromatography of Essential Oils

The effect of drying is quite significant on the overall hop oil content with losses accounting for 30–40% [41] and are mainly associated to the high water vapor volatility [42]. Table 5 provides a summary with standard deviation values of the essential oils namely myrcene, linalool, β-caryophyllene and humulene analyzed through gas chromatography for 25 kg/m² bulk dried at 60 °C and 65 °C, respectively.

Table 5. Essential oils with standard deviation (SD) prior to and after drying process extracted using GC-FID.

| Temperature [°C] | Essential Oils [g/kg hops] | | | | | | | |
| | Myrcene | | Linalool | | β-Caryophyllene | | Humulene | |
	Fresh	Dried	Fresh	Dried	Fresh	Dried	Fresh	Dried
60	4.52	4.17	0.09	0.09	0.72	0.73	2.35	2.45
SD	0.90	1.30	0.01	0.08	0.21	0.35	0.71	1.16
65	7.30	5.09	0.14	0.10	0.76	0.75	2.47	2.54
SD	4.84	0.81	0.02	0.004	0.08	0.06	0.33	0.20

In line with the results on hop oil yield (Section 3.3.1), large deviations in the essential oils is observed. As mentioned previously, hops are a heterogeneous product and the aroma/essential oil content significantly varies within and between the bulks. Furthermore, as mentioned previously the environmental conditions during harvest (dry, dew or rain wet) between the repetitions also varied significantly. Factors such as environmental conditions and harvest time, influences the amount on essential oil components within hops [43]. With harvest lasting for less than two weeks, variations in harvesting conditions during this period can further add to the large variations in oil components [44]. In additions to these factors, the overall sample size was small and hence large deviations were observed. Thus, making it a poor estimator for representation of deviations within the samples. Figure 9 represents the myrcene content prior to and after drying for 60 °C and 65 °C respectively. Losses in myrcene content after drying are observed in both cases. The losses are relatively higher for 65 °C as compared to 60 °C.
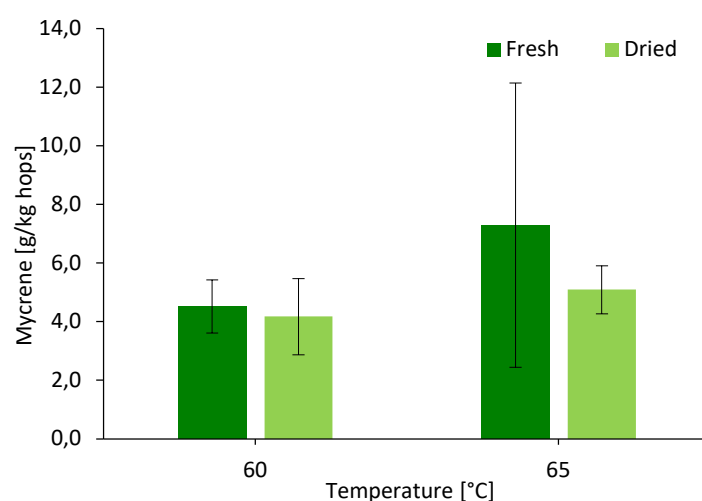

Figure 9. Results obtained from gas chromatography analysis for myrcene in g/kg hops at 0 min and 210 min for hop cones dried at 60 °C and 65 °C at 0.35 m/s.

Myrcene is a terpene hydrocarbon and is one of the most important and sensorially dominant component of fresh hop cones [41]. It is a key contributor to the hop aroma and encompasses 17–37% of the total hop oil [45]. During the drying process, myrcene undergoes oxidation and polymerization which leads to cyclic reactions that form products such as terpenoids which include linalool, and geraniol [46]. Furthermore, myrcene is highly volatile at high temperature. With hop drying

temperatures ranging between 55 °C and 65 °C, significant losses of myrcene are expected. Studies conducted on essential oil losses show that myrcene losses range between 25% and 30% [44,47]. Within the present study myrcene losses of 7.74% and 30.27% were observed for 60 °C and 65 °C, respectively. Experimental investigation conducted on Laurel (*Laurus nobilis* L.) leaves also reveal losses in monoterpene hydrocarbons such as myrcene due to volatility with steam or potential chemical rearrangement of compounds [39].

Linalool is one of the key contributors to the hoppy flavor within beer. It provides a citrusy, flowery and fruity aroma to the beer [4,48]. During the drying process linalool content also undergoes significant changes but unlike myrcene, the losses associated to the linalool are much lower [47]. The results from GC analysis (Figure 10) reveal that after drying at 60 °C, no significant losses in the linalool content were observed. However on increasing the temperature to 65 °C, the losses were much higher as compared to 60 °C. Investigation conducted by [47] show that increasing the temperature increases the losses in linalool content. Too high temperatures such as 80 °C or 90 °C have shown to increase the linalool content. This increase is associated to presence of glycosidically bound linalool that are still active at high temperature and indirectly increase the linalool content. Low temperatures such as 50 °C have shown to retain maximum linalool content as compared to those at 65 °C [47]. Similar losses in linalool content were observed for the Mandarina Bavaria variety dried at 60 °C [17].
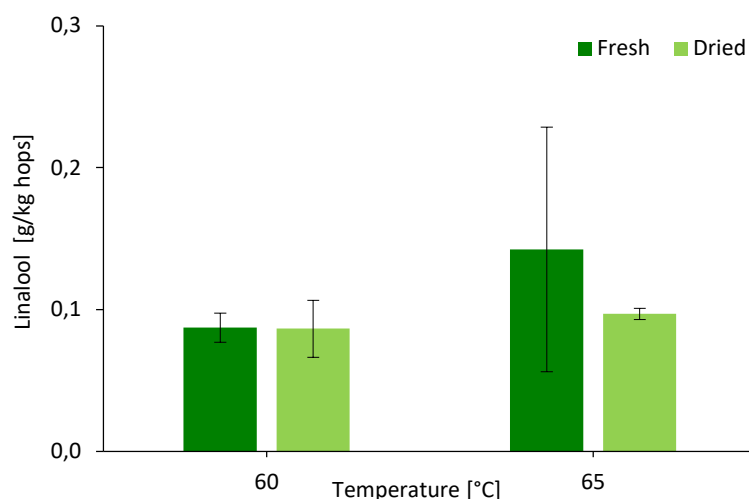

Figure 10. Results obtained from gas chromatography analysis for linalool in g/kg hops at 0 min and 210 min for hop cones dried at 60 °C and 65 °C at 0.35 m/s.

β -caryophyllene and humulene belong to the sesquiterpenes group and are both hydrophobic in nature; thus have limited solubility in aqueous solutions such as beer [46]. During the drying process, both compounds undergo oxidation and form various other products. Oxidation of β-caryophyllene leads to formation of caryophyllene oxide, 14-hydroxy-b-caryophyllene, caryolan-1-ol,4S-dihydrocaryophyllene-5-one, (3Z)-caryophylla-3, 8(13)-diene-5a-ol, caryophylla-4(12), and 8(13)-diene-5a/b-ol [46,49]. The dominant products formed are caryophyllene oxide and 14-hydroxy-b-caryophyllene that have musty, floral, spicy, cedar, woody odor. Humulene theoretically oxidizes to three humulene monoepoxides however only two, namely mono and diepoxides, are found to be present in hops and have hay-like, moldy, cedar like odor [46]. From the results obtained for β-caryophyllene (Figure 11) and humulene (Figure 12), the values after drying are observed to have remained either similar or increased as compared to fresh hops. As mentioned previously, the Hallertauer Tradition has a typical herby, spicy aroma and oxidation of β-caryophyllene and humulene could have led to the potential increase of the byproducts of these main compounds. Thus, explaining the corresponding increase in the β-caryophyllene and humulene content after drying.
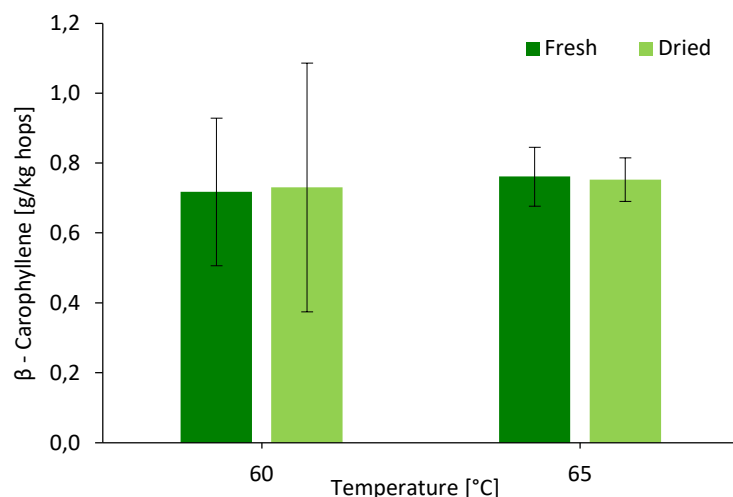
Figure 11. Results obtained from gas chromatography analysis for β-caryophyllene in g/kg hops at 0 min and 210 min for hop cones dried at 60 °C and 65 °C at 0.35 m/s.
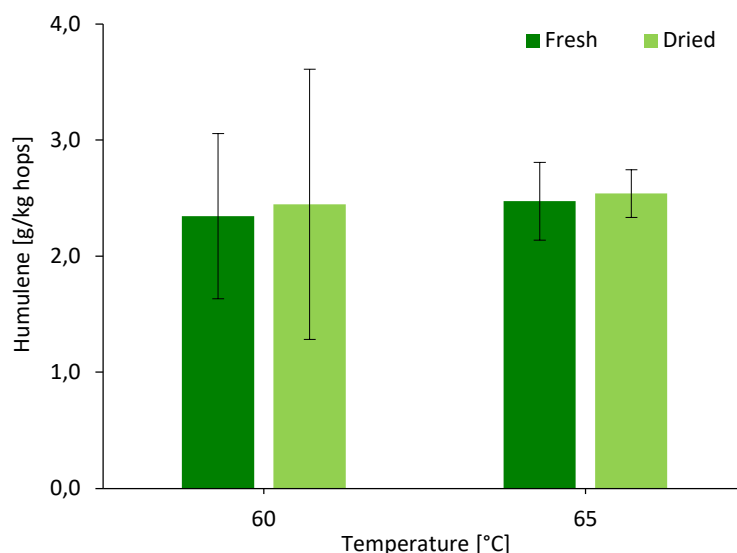

Figure 12. Results obtained from gas chromatography analysis for humulene in g/kg hops at 0 min and 210 min for hop cones dried at 60 °C and 65 °C at 0.35 m/s.

3.4. Color Development

Figure 13 provides the results for the CIE L*a*b* values measured using a chromameter and the values obtained from processing the acquired RGB images. R^2 values of 0.96, 0.90 and 0.95 were obtained for L*, a* and b* respectively. However, significant differences in the actual values were observed especially in case of L* values. The illumination system used for the RGB imaging was observed to have lower intensity as compared to the illumination within the chromameter and could potentially have affected the lower values from the RGB camera system.
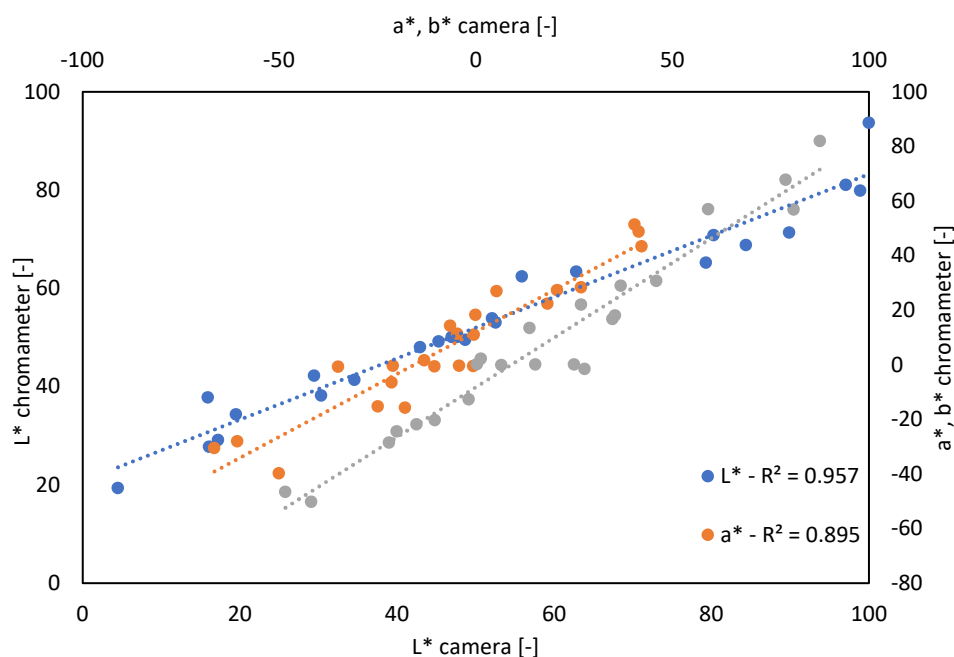
Figure 13. Correlation between CIE L*a*b* values between chromameter and RGB camera system.

The color of fresh hop cones varies between slight dark green to yellow green on the exteriors depending on the variety and harvest time. During the drying process, these hop cones undergo discoloration due to influencing factors such as drying time, temperature, and air velocity. The results obtained for the change in color during the drying process under various conditions is presented in Figure 14.
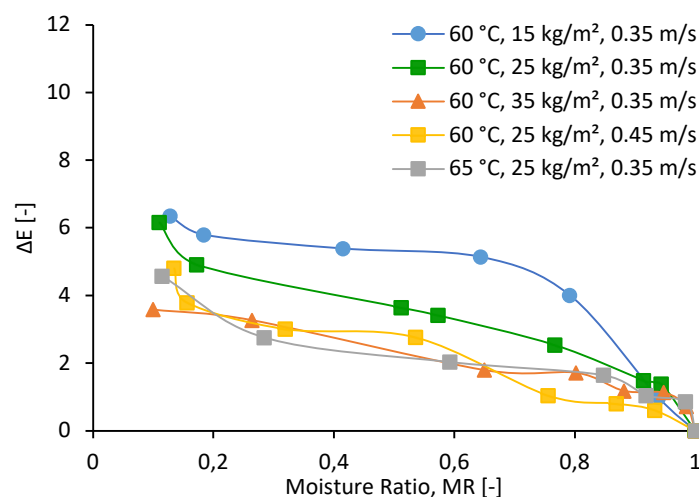

Figure 14. ΔE as a function of moisture ratio (MR) for hops dried under different process settings and different bulk weights.

The highest ΔE values were observed for hops with bulk weight of 15 kg/m² dried at 60 °C and the lowest ΔE values for hops with bulk weight of 35 kg/m² dried again at 60 °C. The associated discoloration is caused during the initial drying phase when the hop cones are colder than the surrounding air. Thus, leading to recondensation of water on the top layers of the hop stack [8,50]. Furthermore, the formation of compact zones or nests in the 15 kg/m² bulk led to uneven drying of hop cones which in turn increased the required specific drying time. As drying time is also an influencing factor for color changes, the longer drying time combined with lower bulk weight, could

have led to the observed discoloration in the 15 kg/m² bulk. The overall results for the total change in color is in contradiction to the results obtained by [3]. According to investigation performed on Mandarina Bavaria with bulk weight of 12, 20 and 40 kg at 60 °C, the highest color changes were observed for 40 kg bulk and lowest color changes for 12 kg bulk [3]. The contradiction between the two studies could potentially be due to two factors. Firstly, the RGB system set up within this study was outside the drying chamber as compared to the system setup of [3] wherein the camera was placed above the bulk within the drying chamber. Secondly, the study conducted by [3] considers the variety Mandarina Bavaria while the current study focuses on the variety Hallertauer Tradition. As each variety has slight differences in color as well as the initial moisture content values, it is possible that opposite results are obtained for experimental investigations performed using the same equipment but slight parameter variations. Furthermore, the varying cone size and the natural heterogeneity in hop cone color could have also influenced the color measurements.

Comparing the results for 25 kg/m² bulk dried at 60 °C and 65 °C, it is observed that the ΔE values for samples dried at 65 °C are lower than those dried at 60 °C. Drying time and drying temperature are two of the influencing factors with degradation of color [51]. An increase in either of two factors can lead to higher degradation of color. The exposure time for the hop cones dried at 65 °C was lower than that of those dried at 60 °C. Investigations on 3 mm thick carrot slices have shown maximum degradation at 50 °C where the exposure time is higher and are concurrent to the findings from the current study [30]. Increasing the velocity has also shown to reduce the color change within the hops. Similar results for apple drying under increased velocity were also obtained [20].

Drying temperature has also shown to influence the lupulin color within the hops. Under optimum conditions, the color tends to remain lemon yellow while at higher temperature and longer exposure time the color degrades to brown [52].

3.5. Energy Consumption

As shown in Figure 15, the highest specific energy consumption was required by the 15 kg/m² bulk (113476 kJ/ kg$_{H2O}$), followed by the 25 kg/m² bulk (91955 kJ/ kg$_{H2O}$) and finally the 35 kg/m² bulk (84794 kJ/ kg$_{H2O}$). The 15 kg/m² bulk required 180 min for drying, while the 25 kg/m² and 35 kg/m² bulk required 210 min and 240 min, respectively. During the drying process for the 15 kg/m² bulk, air pockets were observed to have formed after the first 60 min. These air pockets might have caused the hops cones to clump in one section and thus requiring a much longer time to dry. For 25 kg/m² and 35 kg/m², the overall bulk weight and the height of the bulk dissuaded the formation of air pockets during the drying process and hence required comparatively lower energy to dry the hop cones.
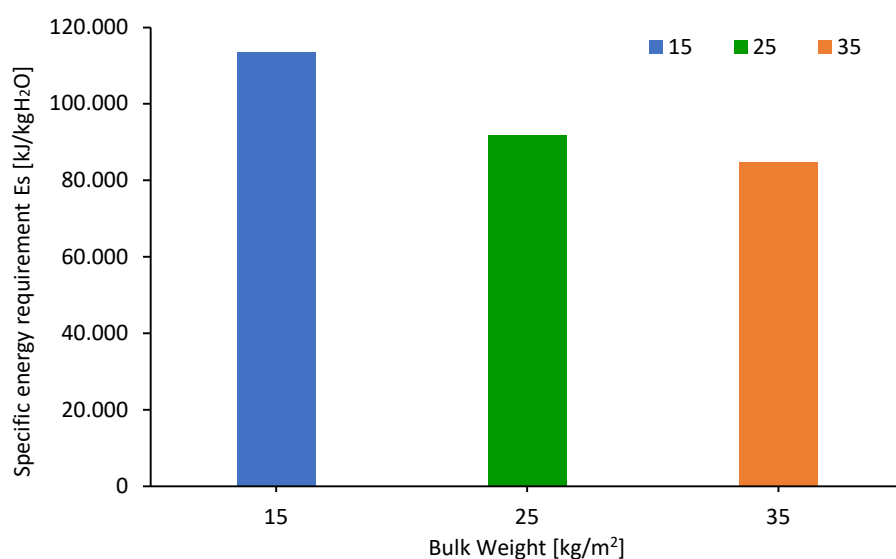

Figure 15. Specific energy requirement for three different hop bulks dried at 60 °C and 0.35 m/s.

On increasing the velocity of the 25 kg/m² bulk to 0.45 m/s, the overall energy consumption is also observed to increase as compared to 0.35 m/s. The difference between the drying time is insignificant and, thus, does not drastically affect the consumption values. The factor influencing the increased energy consumption in this case is the velocity. Results of energy consumption with varying velocity is presented in Figure 16a.
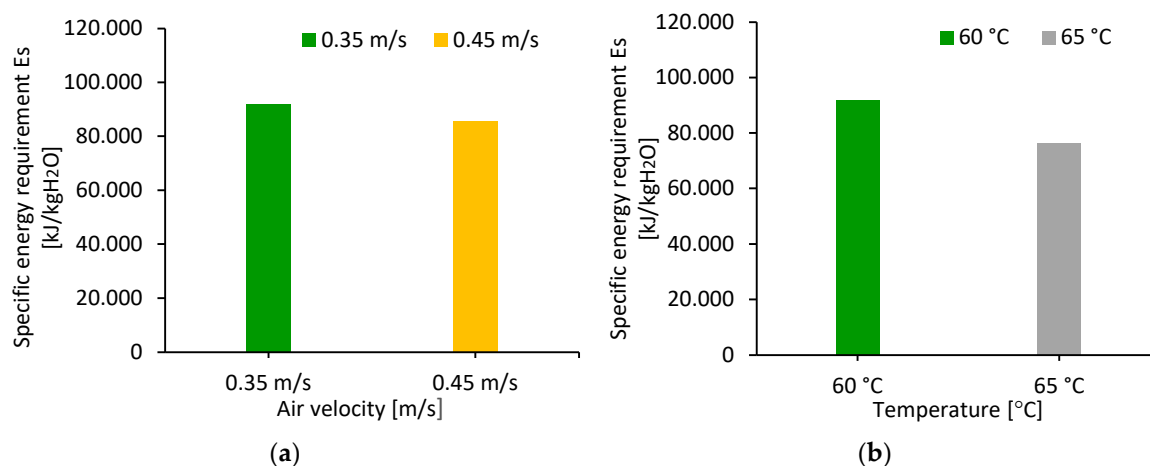

Figure 16. Specific energy requirements for 25 kg/m² bulk dried at (**a**) 0.35 m/s and 0.45 m/s at 60 °C (**b**) 60 °C and 65 °C at 0.35 m/s.

Lower energy consumptions were observed for 65 °C (Figure 16b). The decrease in drying time for higher temperatures compensates for the overall energy demand. The effect of temperature was observed to have a significant influence on the specific energy consumption and is in agreement with previously conducted investigations by [23,53].

4. General Discussion

This study investigated the effects of varying bulk weights and process settings on the drying behavior and product quality attributes such as color, hop oil yield, hop oil components and energy consumption. The study also extended to analyze the influence of hop cone size on the overall drying behavior and its extended effects on quality.

Results obtained from the drying curves show that low temperature in combination with high bulk weight lead to an increase in the overall drying time. Increasing the velocity or the temperature does not significantly influence the drying behavior. Factors such as the environmental conditions during harvest as well as during the drying process, the hop variety in focus and the cone size distribution influence the overall drying behavior. Results obtained from classifying hop cones by size reveals that for the variety Hallertauer Tradition, the majority of the cones lie between small to medium size. The results reveal that such distribution of cone can lead to increase in the air resistance, void fraction and hence increase the pressure drop. In case of 15 kg/m² bulk, this further led to the formation of air channel which affect the moisture content distribution within the hop stack. Thus, the 15 kg/m² bulk requiring a comparatively longer specific drying time than expected. Hop cone size is a natural parameter and varies according to the variety in focus. In order to reduce the resistances, considering bulk density rather than bulk weights/height has been observed to be a better solution [3,9,54].

In terms of color changes, the highest discoloration of ΔE = 6.83 was obtained for the 15 kg/m² bulk, followed by the 25 kg/m² at ΔE = 6.16. The lowest discoloration was observed for the 35 kg/m² bulk at ΔE = 3.58. The higher discoloration of the 15 kg/m² bulk can be associated to the formation of nests or compact zones which increased the required amount of drying time. As temperature and drying time are influencing factors for color changes, an increase in either of these factors, can cause significant effects on the color quality of the product. Temperature and time are also influencing factors for the specific energy consumption, with the 15 kg/m² bulk consuming the high amount of

energy per unit of product. In order to optimize and reduce the energy costs, it is important to consider alternative solutions that can be implemented with the current drying techniques [9].

Drying temperature is not only an influencing factor affecting color, but also affects the yield and chemical compositions within hops. The drying process has a significant effect on the hop oil yield and composition, and thus a decrease in the values after completion of drying is expected. On increasing the temperature from 60 °C to 65 °C, increased losses for yield were observed. This is analogous to the results of previous studies [3,17]. In terms of composition, the expected trend of losses was observed for myrcene and linalool, however, an increase in β-caryophyllene and humulene was observed at higher temperature of 65 °C. The increase in β-caryophyllene and humulene was previously observed in storage of hops for 24 h prior to drying wherein it was believed the formation of other by products led to an overall increase in these components [17]. As no storage tests were conducted within the current study, the results obtained for β-caryophyllene and humulene are in contradiction. However, hops in general have a large heterogeneity in the aroma components which fluctuate based on various conditions such as hop variety, hop garden, soil, environmental factors at harvest, etc. [44]. The increase in the β-caryophyllene and humulene content could potentially also be associated to the other influencing factors.

5. Conclusions

The investigations conducted within the current study demonstrated that process and product quality in hop drying depend on multiple factors such as harvest time, drying conditions, and bulk characteristics (weight and cone size distribution). An optimization of the process would require consideration of multiple potential target conflicts, i.e., energy demand vs product quality, reduction of drying time with bulk reduction vs additional labor demand and machine times caused by additional harvesting schedules.

The study, therefore, indicates the need for development of systems that consider various factors such as hop variety, harvest maturity, environmental conditions at harvest (dry, rain wet) and bulk quality (e.g., cone size distribution), for optimum process settings. This includes the necessity for the development of non-invasive measurement systems in combination with integration of adaptive control systems. With advances in optical sensors, computational models and with the advent of internet of things (IoT) integrated processing systems, it will be possible to further develop tailored systems which consider all above aspects. In addition to integration of measurement and control systems, the study also highlights the need for further work with bulk density rather than bulk weight/height in combination with pressure drop measurements as this will not only aide in optimizing the process parameters but also help in reducing the energy consumption and, thus, the associated processing costs.

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