In-process investigation of the dynamics in drying behavior and quality development of hops using visual and environmental sensors combined with chemometrics

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\section*{A B S T R A C T}

Hops are a key ingredient for beer brewing due to their role in preservation, the creation of foam characteristics, the bitterness and aroma of the beers. Drying significantly impacts on the composition of hops which directly affects the brewing quality of beers. Therefore, it is pivotal to understand the changes during the drying process to optimize the process with the central aim of improving product quality and process performance. Hops of the variety Mandarina Bavaria were dried at 65 °C and 70 °C with an air velocity of 0.35 m/s. Bulk weights investigated were 12, 20 and 40 kg/m² respectively. Drying times were 105, 135, and 195 and 215 min, respectively. Drying characteristics showed a unique development, very likely due to the distinct physiology of hop cones (spindle, bracteole, bract, lupulin glands). Color changes depended strongly on the bulk weight and respectively. Drying characteristics showed a unique development, very likely due to the distinct physiology of hop cones (spindle, bracteole, bract, lupulin glands). Color changes depended strongly on the bulk weight and re-

\section*{1. Introduction}

Hop cultivation has a long tradition in Europe, with first mentions dating from around 1000 years ago \citep{Hirschfelder and Trummer, 2016}. Initially, hops were primarily used to increase the microbiological stability of the beers which helped increase the shelf-life and allowed for long distance transport. Over time, hops have increasingly been used for other purposes, such as creation of specific foam characteristics, bitterness and aroma of beers \citep{Rybäček, 1991; Hirschfelder and Trummer, 2016}. Whilst bitterness is still the most important motivation for selecting specific types of hops, aroma and flavor hops have received new attention due to recent trends in craft beer manufacturing.

Historically, the hop markets were dominated by bitter and high α acid hops, accounting for 75–80% in the US market, for example. According to the Barth report \citep{Barth, 2018} in 2017, however, the market shares between bitter and aroma hops in the US and Germany were 23.1 and 76.9% and 56.7 and 43.3% respectively. This shows a clear shift in market demands towards craft beer production. In this context, it is worth noting that the specific hop demand in craft beer brewing is significantly increased, which is partially due to the addition of the cold hopping process during the fermentation stage.

Besides the variety, the optimum harvesting date depends on a multitude of environmental factors, such as environmental temperature, precipitation, light, location, soil quality, and exposition.
According to Münsterer (2017a, b), the time frame between cone formation and harvest maturity is 4 weeks (minimum 25 days) for α hops, 5 weeks (minimum 30 days) for high α hops and 5 weeks (minimum 30 days) for flavor hops. At optimum harvest maturity, cone color changes from dark green to light green and yellowish. Münsterer (2017a, b) further found that the dry matter content is a good indicator for the determination of maturity. The timing of harvest has a significant impact on both the overall oil content and its composition. Generally, the later the harvesting date the more pronounced the aroma expression. However, towards the end of the ripening stage, hops increasingly develop an oniony and garlicky aroma due to an increase in sulfur compounds (Cocuzza et al., 2013; Lutz et al., 2009; Münsterer, 2017a).

Fresh hop cones are highly susceptible to rapid degradation and have to be processed within 4–6 h after harvesting to retain optimum quality (LfL, 2019). The most commonly found preservation method for hops is convection drying during which the fresh hop cones are dried from between 78 and 84% (LfL, 2019) to 10% desired final moisture content, usually in multi-stage kiln dryers.

Bitterness is primarily created by non-volatile components, such as α and β acids, which are secondary metabolites of the hops and are responsible for the bitterness of beers (De Keukeleire, 2000; Nagel et al., 2008). They mainly accumulate in the lupulin glands which lie between the braces of the cones (Priest and Stewart, 2006) and on the underside of young leaves (Kavalier et al., 2011). During storage, α acids in particular are susceptible to degradation. These changes can be expressed using the Hop Storage Index which was established as a parameter for the evaluation of the freshness of hops and hop pellets. Cocuzza et al. (2013) showed in their research that the harvesting date also has a significant influence on the Hop Storage Index of fresh hops. Forster and Beck (1985) found that α acid losses are between 3.5 and 10% during drying, depending on the overall quality of the hops. Zeisig (1970) showed that an increase of loss by 5% occurred when the drying temperature was increased from 55 °C to 65 °C at constant air velocity and bulk height. This is potentially related to a lack of sufficient air throughput in the transportation of the evaporated water and the bulk height within the dryer.

Like bitter components, aromatic components are located in the lupulin glands. Essential oils make up 0.5–3% of the dried hop weight (Sharpe and Laws, 1986; Steinhaus and Schieberle, 2000). In contrast to the bitter components, which are mainly affected by degradation due to high water activity levels over an extended period of time, aromatic compounds and essential oils are highly volatile and susceptible to degradation before, during and after the drying process (Aberl, 2016). During drying, these losses are mainly due to water vapor volatility (Münsterer, 2017b). More than 400 volatile components are known in hops. The highest percentage of essential oils are carbon hydrates which are responsible for 69% of odor activity (Steinhaus and Schieberle, 2000). Reactions of aromatic components with oxygen are responsible for around 34% of the odor activity (Steinhaus and Schieberle, 2000). Sulphuric compounds such as 4-MMP are highly aroma active despite them being only present in very low concentrations.

Myrcene and linalool are seen as key substances for the overall aroma (Steinhaus and Schieberle, 2000). Myrcene’s smell is associated with geranium while linalool is described as sweet, flowery and citrusy. The taste threshold for aromatic compounds is in the region of few µg/l beer (Kurzweiler et al., 2013). According to Aberl (2016), drying of hops results in a loss of hop oils of 30–40%. Myrcene is highly volatile at temperatures usual for hop drying operations. Kaltner (2000) and Kammhuber (2018) reported losses between 25 and 30 % during drying, however it was also pointed out that hop aroma quality tended to increase with a decrease in myrcene concentration.

Numerous previous studies on drying of sensitive agricultural products, including hops, have shown that the impact of air velocity, and thus specific air mass flow, needs to be considered when evaluating optimization potentials for the process (Arul and Bese, 2016; Ashitani et al., 2018; Fatouh et al., 2006; Kaya and Orhan, 2009; Münsterer, 2015; Orphanides et al., 2017; Sturm et al., 2012; Hermanek et al., 2016; Münsterer, 2016). In practice, air humidity regularly reaches 100% at the start of the drying process, which has direct negative effects on the quality of the hop cones exposed to these conditions. Firstly, the water which is transported to the surface of the cones cannot be removed from the surface, which leads to color degradation. Secondly, these cones are highly likely to be colder than the surrounding air, which in turn leads to a re-condensation of a proportion of the water in the air which further increases discoloration (Münsterer, 2015; Sturm et al., 2016). The knowledge of product temperature throughout the process has also proven to provide valuable information for the optimization of the process and has been linked to the development of discoloration of the product and transition from one drying phase to another for products such as apples (Fiorentini et al., 2015; Sturm et al., 2014).

Another aspect heavily impacting the final product quality, the overall energy demand and efficiency is the point in time when the drying process is terminated. This is, to date, largely a result of the experience of the processor. However, a uniform result cannot necessarily be reached (Hermanek et al., 2018), which also has direct consequences for the subsequent conditioning phase (Münsterer, 2006, 2012; Rybka et al., 2019). Due to their structure, the leaves of the hop cones dry significantly quicker than the spindles (Hermanek et al., 2018), leading to a product which is not in an equilibrium state. Commonly, the leaves have a final moisture content of 6–8%, while the spindle remains at 12% (Münsterer, 2006, 2012). Thus, conditioning is an inevitable further processing step.

Currently, temperature and humidity sensors are usually placed within the hop stack during drying. This method works well as a local (in spatial terms) non-destructive method to determine hop moisture content. However, large variations in moisture content may exist within the overall hop stack (Münsterer, 2006). This method also ignores any potential to continuously monitor the dynamics of changes within the product which go beyond water content. In recent years, development of dryers has been strongly focused on the inclusion of product relevant information, with the aim of the realization of smart drying systems which not only measure these changes non-invasively but also use these as input variables for process control (Martynenko, 2017; Martynenko and Misra, 2020; Martynenko and Sturm, 2019; Sturm, 2018; Sturm et al., 2018). To gain a deeper understanding of the dynamic processes within the drying of plant based products, such as hops, information from non-invasive measurement devices such as color cameras (CCD), spectroscopy and hyperspectral imaging (HSI), as well as thermography, can be combined with product related information from chemical and textural analysis. With machine learning methods becoming more sophisticated, computing power increasing rapidly and prices falling, new ways of dealing with data and developing drying systems based on the principles of Quality by Design are emerging, i.e. using the raw material characteristics and change dynamics as basis for control development and execution (Martynenko, 2017; Sturm et al., 2018).

Non-destructively monitoring chromaticity and moisture content, as well as the changes in chemical composition, over the entire hop stack could bring great benefits to the hop drying industry. HSI offers a potential single solution to investigate both chromatic and chemometric changes in the hops continuously during drying (Crichton et al., 2016; Crichton et al., 2018; Pu et al., 2015; Pu and Sun, 2015; Su et al., 2018; Wu et al., 2012, 2013), while color imaging and thermography can deliver further valuable information on the changes a product undergoes during drying (Martynenko, 2006; Martynenko and Kudra, 2015; Martynenko, 2017; Sturm, 2018; Sturm et al., 2014). Several previous studies have also shown, that HSI in the visible and near infrared areas is suitable for the determination of phenolic and other bioactive compounds in plant-based products (Gaston et al., 2010; Huck, 2015; Yang et al., 2015). Combined with other process data and their fusion, a
better understanding of the dynamics of quality changes within hops during drying can be gained. Based on this information, optimized process settings and dryer set-ups can be realized.

In this study, for the first time, a full array of visual sensors was integrated into a pilot scale drying system to investigate the dynamic changes of selected quality criteria, namely color, Hop Storage Index, α and β acids, product and air temperature, as well as air humidity, throughout the drying process, with different bulk weights and drying temperatures. These criteria were then fused with the results of color and HSI to predict water content and distribution thereof. Furthermore, selected chemical components, i.e. myrcen, β-Ocimen a and 1-octen-3-ol, were chemometrically linked to HSI. Conclusions for optimized hop drying were drawn from the combined data. The study is a proof of concept for integration of non-invasive measurement systems into fully operational small-scale commercial driers and, thus, lies the foundation for the realization of intelligent drying systems for sensitive agricultural produce.

2. Materials and methods

Trials were conducted at the Research Station Hüll, Hallertau, Germany on five consecutive days in September 2015 to minimize variations in raw material quality due to harvest dates (Cocuzza et al., 2013).

2.1. Materials

Hops of the variety Mandarina Bavaria were investigated. Mandarina Bavaria is a hybrid between Cascade and a male Hüll breeding strain (Lutz et al., 2013), and is described as less hop-typical, but with a pronounced fruity and exotic aroma of mandarin and orange (Lutz et al., 2015).

All raw materials originated from the same hop garden close to the research station. Hops were harvested one hour before drying tests were conducted. Hop cones were separated from the vines using a commercial de-vining machine (Hopfenpfückmaschine Wolf WHE 220), weighed and then transferred into a pre-heated dryer and drying tests were started immediately.

2.2. Methods

2.2.1. System set-up

A pilot plant scale dryer with a drying area of 1 m² and an integrated heat recovery system was used as described by Hofmann et al. (2013). The system was modified by the introduction of an RGB camera, a hyperspectral imaging system and the respective lighting installations as described by Crichton et al. (2016).

The dryer was further equipped with an array of temperature, humidity and air flow sensors to assess the temporal and spatial conditions within the system. Combined temperature/humidity probes (Testo 174H, Testo, Lenzkirch, Germany) were installed and positioned at different filling heights within the bulks to measure the development of temperatures and air humidity throughout the bulks. Product temperature at the surface of the bulk was measured continuously using a pyrometer (Bartec Type R300-123). All values, except of the combined temperature/humidity probes, were transferred directly to a computer and stored.

2.2.2. Sampling

Standard process settings used were 65 and 70 °C drying air temperature and 0.35 m/s air velocity. For the simulation of real conditions, the bulk weight of the samples was used rather than the bulk height. Three different bulk weights (12, 20 and 40 kg/m²) were investigated at 65 °C, resulting in 25, 30 and 33 measurements across the triplicate experimental run. Furthermore, at 70 °C for 0.35 m/s 22 measurements were conducted and for the purpose of hyperspectral measurements an additional 24 samples were taken at 0.15 m/s. Air temperature, product temperature, relative humidity, color, and spectral characteristics were measured. All investigations were conducted in triplicate.

Samples were taken at the start of the drying process, after 15, 25, 35, 45, 75 min and then every 30 min thereafter until the samples reached storage moisture content. RGB and hyperspectral images were taken directly before the samples were removed from the dryer. Image capture at these set time intervals across the 4 different drying conditions led to the capture of 134 hyperspectral images and 134 corresponding calibrated RGB images. Three hyperspectral images were subsequently discarded due to capture error, so all subsequent analysis was performed upon 131 images. The test train split was carried out on a 2:1 basis, resulting in a 88:43 basic train test sample split. The calibration of the system is detailed in Sections 2.2.3 and 2.2.4 and within Crichton et al. (2016).

Sampling was conducted on the surface of the bulk underneath the HSI system and the amount removed was replenished from other areas of the surface (1 m²). For sampling, a beaker of 2000 ml capacity was used and for each sample the equivalent of 1600 ml (mlw) was removed. A small proportion was then used for dry matter assessment (24 h, 105 °C) (AOAC, 1995) whilst the rest (ca. 1200 mlw) was vacuum packed and immediately frozen at −20 °C for chemical analysis. All tests were conducted in triplicate.

2.2.3. RGB imaging

The RGB imaging system was directly integrated into the drying chamber (Fig. 1). To prevent overheating to the unit, a permanent pressurized air blast was provided, and the camera temperature continuously measured using a thermocouple. The maximum device temperature was 31 °C. A color DFK 72AUC02-F (The Imaging Source, Germany) of a resolution of 1944*2592 (H*W) was used. Illumination was provided by 2 Philips fluorescent light sources (Philips, Netherlands) of CCT 6500 K and luminance of 2100 Lumens each. Color calibration of the RGB camera using polynomial color correction (Finlayson et al., 2015) was carried out. An X-Rite Color Checker Classic 24 patch chart (X-Rite Inc., Michigan, USA) was imaged using a set exposure under imaging conditions. Colorimetric data of each patch was then measured using a calibrated spectrometer (Q mini, RGB Photonics GmbH, Kelheim, Germany). Calculated XYZ values were then converted into CIELAB co-ordinates.

To carry out the calibrated CIELAB capture correctly the halogen light source was turned off prior to RGB image capture (leaving only the 6500 K fluorescent sources on).

2.2.4. HSI set-up, imaging, calibration and statistical analysis

To enable the imaging of hops during drying, a Specim V10E hyperspectral camera (Specim Spectral Imaging Ltd., Finland) was placed within the dryer inside a protective casing above the hop stack (Crichton et al., 2016). The camera was used in combination with a mirror translation unit and a 35 mm Schneider lens (Xenoplan 1.9/35, Schneider Optische Werke GmbH, Germany). Illumination was provided using a halogen security lamp fitted with a 600 W IP65 rated halogen lamp, which was attached on the interior wall of the dryer and focused at the region directly beneath the camera system (Fig. 1). The protective casing was constructed of steel, with a rockwool insulation lining of 30 mm thickness. The casing also had a viewing window covered with a clear acrylic window sheet (Fig. 1). This allowed the camera to view the hops situated directly below. This protective casing also provided ventilation to the camera during operation to prevent overheating. Temperature within the casing was measured continuously using a thermocouple. The maximum temperature within the casing was 27 °C. This protective casing was attached to the interior of an experimental dryer above the top drying tray as outlined by Hofmann et al. (2013).

The lens was of a manual focus type and was focused at a specific
hop depth, which led to an increase in blur towards the lowest hop depths. Hyperspectral cubes were recorded using the Specim SpectralDAQ software (Specim Spectral Imaging Ltd., Finland), with all processing occurring within Matlab using software previously utilized in Crichton et al. (2015) and Crichton et al. (2016). Noise removal was carried out in the same manner as presented in Crichton et al. (2015).

Due to the nature of product shrinkage during drying, the height of the hops changed which resulted in changes in the light intensity over the area. To take these changes into account, the measurement of the incident illumination at a number of different heights was carried out. The resulting illumination field was smoothed with a small window operator to remove the influence of the non-perfectly uniform surface of the white reference tile. This height dependent illumination data was then utilized in combination with hop height measurements carried out at each sample period. Fig. 2 below illustrates the increase in light intensity at the 669 nm wavelength as drying occurs. Each of the 3 columns from left to right shows that as drying occurs, and the depth of hop bed decreases the intensity variation across the viewing field changes. This increase in intensity as the hop moves further from the reference point occurs due to the focal point of the light source. The upper row illustrates the reference white image for each of the hop bed depths, with the lower plots illustrating the intensity map across the white reference in the picture. Images were then processed with the corresponding white reference image to produce the reflectance image on a pixel-by-pixel basis.

The nature of the imaged hops and singular illumination direction led to challenges in image processing, which required further use of preprocessing methods. Both factors meant that, when hops across the region of interest were analyzed, a great variation in spectral intensity existed. In order to counteract this, two different normalization preprocessing methods (Amigo et al., 2013) were used; Normalization and Standard Normal Variate (SNV). Using these two methods had the effect of minimizing the baseline variation between pixels related to hop surface topology, as seen in Fig. 3 below.

After the retrieval of the average pre-processed reflectance spectra from each image, Partial Least Squares Regression (PLSR) analysis was performed upon the spectra against the measured moisture content, and CIELAB chromatic co-ordinates retrieved from the calibrated RGB images. PLSR analysis was performed upon the normalized and SNV data. PLSR is the most common method for investigating the feasibility of metric prediction in combination with spectroscopy and hyperspectral imagining. It attempts to uncover whether any of the retrieved wavelengths vary in a linear manner with respect to the metric investigated. Previous investigations have looked into moisture prediction (ElMasry et al., 2007; Huang et al., 2014; Pu et al., 2015), chromaticity (Iqbal et al., 2013; Wu et al., 2012), maturity (Rajkumar et al., 2012) and pH levels (He et al., 2014) amongst many other metrics. Monte-Carlo uninformative variable elimination (MCUVE) (Centner et al., 1996) and the competitive adaptive reweighted sampling (CARS) (Li et al., 2009) are different wavelength reduction methods, noted from here onward as MCUVE–PLSR and CARS–PLSR, respectively. In some cases, they are good alternatives to the PLSR prediction model (Amigo et al., 2013).

For the statistical investigations, the measured hops were split into the calibration and test sets on a random 2:1 ratio, on the basis of replicate number. Furthermore, each model was cross validated five-fold to ensure all combinations of calibration and test sets were tested.

2.2.5. Modelling of drying curves

Weight and color changes were measured intermittently, while process and product temperature, as well as relative air humidity, were measured continuously. To ensure a meaningful correlation between those values, the modelling of the drying curves based on the measured values was necessary. Two drying models, the Page model (Doyraz, 2004) and the Henderson & Pabis model (Doyraz, 2004), were used to fit the drying curves for the experimental moisture content of hops – Eqs. (1) and (2) below represent the Page and Henderson & Pabis Model respectively.

\[ MR = \exp(-Kt^n) \text{ PageModel} \]

where \( K, t \) and \( n \) are the drying constant, drying time in min and the drying exponent respectively

\[ MR = a\exp(-Kt) \text{ Henderson&PabisModel} \]

where \( a \) and \( k \) are drying constants and \( t \) is the drying time in min.

The moisture ratio MR [-] for hops was calculated using Eq. (3):

\[ MR = \frac{M}{M_0} = \frac{M - M_e}{M_0 - M_e} \]

where \( M_{[gw/gdM]} \) is the moisture content at any given time, \( M_0_{[gw/gdM]} \) is the initial moisture content and \( M_e_{[gw/gdM]} \) is the equilibrium moisture content. As \( M_e \) is relatively small compared to \( M \) and \( M_0 \),
(Doymaz, 2004), the moisture ratio was simplified to Eq. (4)

\[ MR = \frac{M}{M_0} \]  

(4)

Non-linear regression analysis was performed using GraphPad Prism 8.0 software (GraphPad Software, 2018). Adjusted \( R^2 \) (\( R^2_{adj} \)), Root mean square error (RMSE) and Akaike Information Criterion (AICc) were used to evaluate the goodness of fit between the predicted and the experimental data for each of the two models. In addition, the basic requirements for the application of nonlinear regression in the form of residual analysis were examined. The following assumptions were tested: Variance homogeneity, normal distribution of residuals and lack-of-fit of the fitted model. The independence of the measurements was ensured by the test design.

A comparison of fits between the two models was also performed using the AICc, rather than F test due to the assumption of non-nested
models. The comparison of fits not only provides a difference in the AICc but also the likelihood a certain model is right on a percentage scale. 

$R^{2}_{adj}$ values rather than $R^2$ values were used for evaluation as these values have been adjusted to the number of predictors in the model. AICc is a model selection criterion that estimates the most likely model for the selected data. These values are not always negative, however, non-linear regression analysis can potentially lead to negative values of AICc, as is the case for the selected hop data (Burnham and Anderson, 2002). For non-linear regression, homoscedasticity or test for appropriate weighting ($P$ value) assumes that ‘average distance from the curve is the same for all parts of the curve’ (GraphPad Software, 2018). Therefore, a model is estimated fit for the data if the values for $R^{2}_{adj}$ and $P$ likelihood are high, RMSE & AICc are low, and homoscedasticity ($P$ value) $> 0.05$.

2.2.6. Chemical analysis and determination of the hop storage index

For the chemical analysis, the frozen samples were freeze dried (LABONCO FreeZone 2.5 Plus). Determination of HSI, α and β acids was conducted according to the EBC Method 7.13 (EBC, 2007). A total of 134 samples were analyzed. Samples were extracted with toluene followed by dilution of the extract in methanol and alkaline methanol. The diluted solution was measured spectroscopically at 275 nm, 325 nm and 355 nm using a Thermo Scientific Eva 201 photo spectrometer. Hop Storage Index and β acids contents were determined using the following Eqs. (5)–(7):

$$\text{HopStorageInd} = \frac{A_{275}}{A_{325}}$$

(5)

$$\alpha = (-19.07 \cdot A_{275} + 73.79 \cdot A_{325} - 51.56 \cdot A_{335}) - F$$

(6)

$$\beta = (+5.10 \cdot A_{275} - 47.59 \cdot A_{325} + 55.57 \cdot A_{335}) - F$$

(7)

For the tests presented $F$ was 0.667. To make the results comparable, the results for all water contents were normalized to 10% final water content.

For determination of aromatic compounds, samples were also freeze dried and afterwards further processed using a statistic headspace-method (Agilent Headspace Sampler 7697 A) developed inhouse with the settings purge gas: He 5.0; loop volume: 3 ml; transfer line diameter: 0.53 mm; transfer line length: 1.00 m; incubation time: 60 min; incubation temperature: 80 °C; injection time: 30 s.

Due to a malfunction of the freeze dryer, which only became visible after the chemical analysis, only 73 out of 134 samples could be used for the model development. The test train split between the sample was 49:24.

3. Results and discussion

3.1. Drying behavior

For all drying conditions there is a characteristic development of drying velocity with a rapid loss of moisture in the initial phase, followed by a phase of reduced drying velocity and another phase of increased moisture loss thereafter before eventually transitioning into a second phase of reduced drying velocity (Fig. 4). This could be due to the structure of the hop cones which consist of a spindle, bracteole, bract and lupulin glands (Fig. 5). It is very likely that the spindle only starts drying once the leaves have reached a certain moisture content and, thus, develop a sufficiently high suction tension to draw the water out of the spindle.

As already described, at the end of the drying process (at 10%) the leaves are over dried to a moisture content of 6–8%, whilst the spindle still has a moisture content of around 12% (Fig. 5). This fact also supports the theory regarding suction tension as a driving force for moisture removal and, thus, the very specific development of the drying curve.

The ratios between spindle and body of the cone depends strongly on the variety in question, and the stage of development of the bulk and the individual hop cones. This in turn has a significant impact on the drying characteristics and the required conditioning time. Münsterer (2015) investigated 7 varieties to determine the ratio which was found to vary from 6.3 to 7.5 % (Hallertauer Magnum) to 10.0–12.0% (Hallertauer mfr.). Furthermore, as demonstrated in Fig. 5, size and shape of individual hop cones can vary greatly. Thus, both the average size, as well as the size distribution, of cones directly impacts on the bulk density and, thus, the fluid dynamic characteristics of the bulk. To ensure a proper drying behavior of the bulk, these differences significantly influence both the duration of the drying process with the associated air conditions (Fig. 6) and the achievable product quality in terms of color retention (Fig. 7).

3.2. Model comparison for drying behavior

Due to the nature of the experimental and system set-up, it was necessary to model the development of moisture content with the goal of conducting a clear observation of product temperature and air humidity development as a function of moisture content.

A comparison of two models (Page and Henderson & Pabis) was conducted for hops dried at 65 °C and at different bulk weights (12, 20 & 40 kg) and for 20 kg bulk weight dried at 70 °C and 0.35 m/s (Table 1). In both scenarios, $R^{2}_{adj}$, RMSE, AICc, Homoscedasticity and % likelihood were used to estimate the goodness of fit. Higher $R^{2}$ and lower RMSE values were obtained for the Page model. However, the difference between the $R^{2}_{adj}$ and RMSE values obtained from the independent models is relatively close to a conclusive prediction. A considerable difference was observed in the AICc and homoscedasticity values with the values ranging from −50.2 to 88.9 and 0.1634 to 0.4302, respectively. The lower the AICc values, the better the conformity of the model (Raut et al. 2019). Furthermore, through the comparison of the fits, the % likelihood is considerably high for the Page model. Thus, based on the values obtained, the Page Model was determined to be the more representative model for hops drying.

3.3. Color changes

Fig. 6 depicts the total color differences ΔE for the samples dried at
An increase in bulk weight impacted the color changes of the different samples significantly. The changes can be represented through linear regression with a high degree of accuracy. The differences between the bulk weights can be directly correlated to the decrease of water removal potential of a given amount of air with increased bulk thickness (Münsterer, 2015; Sturm et al., 2016). This is of particular importance during the first phase of drying. However, an increase of temperature at identical bulk weight, and thus a reduction of specific air flow, leads to a lower initial color change but does not impact on the final value.

Due to the damage in the bulk dried at 65 °C with a bulk weight of 40 kg after harvest in rainy conditions, the batch had to be discarded as the quality was severely affected and the values were removed from the above comparison.

Fig. 5. Exemplary hop cones of different sizes, cross section through a hop cone and individual leaves and spindles.

Different bulk weights and temperatures.

For explanation, Fig. 7 depicts the total color changes \( \Delta E \) for the different weather conditions for 40 kg bulk weight, while Fig. 8 shows the rH of the air above the bulk and product surface temperature.

It is evident that changes in color significantly depended on the weather conditions. Moderately high moisture (morning dew) did not have an impact on final color changes. However, rain wet hop cones deteriorated very quickly with an overall \( \Delta E \) almost twice as high as the other samples. Air exchange evidently was not sufficient to remove the water quickly enough during drying in the rain wet batch, leading to a rapid increase in browning. Furthermore, within the first 45 min of the process, relative humidity over the bulk was continuously at 100%, making drying impossible and the recondensation of water transported through the bulk very likely due to the wet bulb temperature difference (Fig. 8).

It can be clearly seen that, when surface water was present (rain and dew), the initial rH of the air above the bulk was at 100% while the bulk surface temperature remained almost constant. As soon as the rH decreased below 100% and, thus, surface water was removed completely from the hop cones, product temperature increased according to the resulting bulb temperature. These results are in line with the findings of Sturm et al. (2012, 2014) for apples. Thus, showing a direct relationship between environmental conditions and thermal product characteristics. The somewhat erratic development of both the rH of air
The selected wavelengths. The large variation between the full wave-
range. The green peak present at roughly 550 nm (Fig. 9a), known as
this is mind, the PLSR analysis was undertaken in the 500
region from 500 to 1010 nm. The trials were conducted under quasi-
production conditions using halogen lights which have a low irradiance
below 500 nm and as can be seen from Fig. 9a the utilization of this
region would introduce a significant level of noise to a small data set
and be detrimental to model performance. The light light emission issue
is further enhanced by the lower SNR of the V10E sensor in this range.
It can also be noted that the retrieved reflectance information is greater
above 700 nm than in the region between 500 nm and 700 nm. With
this is mind, the PLSR analysis was undertaken in the 500–1010 nm
range. The green peak present at roughly 550 nm (Fig. 9a), known as
the "green gap" (Gundlach et al., 2009) between the absorption peaks
of chlorophyll a and b at 430 and 664 nm and 460 and 647 nm
respectively, is reduced in size as drying occurs. This indicates a change
in anthocyanin concentration, a sub-group of flavonoids, which have an
absorption peak in the region of 530 nm (Kim et al., 2008). Also of great
interest is how the peaks and troughs present in the 700–1010 nm
region are removed as drying occurs. This development might be due to
the change in concentration of other polyphenolic compounds. These
include flavanols, flavan-3-ols, phenolic carboxylic acids and others as
prenylflavonoids and have both a hydroxyl group (O–H) and an
 aromatic hydrocarbon (C–H). The changes could thus be due to the third
overtone of the third overtone of OH and the fourth overtone of CH in
the region of 740 and 760 nm (Amadio et al., 2017; Clement et al.,
2008; Dusek et al., 2016; Kokaly and Skidmore, 2015; Steinhaus and
Schieberle, 2000). The trough at roughly 970 nm is known to be
directly related to the stretching of the H–O–H bonds (Clement et al.,
2008) within water molecules.

The results of the PLSR (Table 3), MUCVE-PLSR (Centner et al.,
1996) (Table 3) and CARS-PLSR (Li et al., 2009) (Table 4) models
developed show that basic prediction of hop moisture content, CIELAB
chromaticity and chemical estimation can indeed be carried out.

Moisture content estimation can be achieved with a lower error
using the normalized retrieved spectra during drying. This produces a
model which uses only 6 wavelengths and achieves a test set r² = 0.95
and RMSE = 0.24 with the MUCVE-PLS model. This level of perfor-
cance can be seen to be useful up until the last stages of drying.
However, the consistent RMSE performance between the calibration
and test sets for the reduced wavelength set shows the importance of
the selected wavelengths. The large variation between the full wave-
length set PLS models, shown in Table 2, does, however, point to a
problem with overfitting to the calibration set, which may have been
cased by any variation in initial hop quality.

CIELAB a⁺ prediction can be seen to be better serviced using the
normalized spectra with the MUCVE-PLS model with test set results of
r² = 0.81 and RMSE = 2.79 using only 5 wavelengths ([620, 665, 902,
912, 1010] nm). Whilst for CIELAB b⁺ prediction, use of the SNV
spectra using the CARS-PLS method led to the best performing model
with test set performance of r² = 0.73 and RMSE = 1.94. This was
achieved using 13 wavelengths.

Moisture content and chromaticity prediction are very useful
quality metrics to allow the monitoring of hop changes during the
drying process. However, hops, in a chemical sense, are also very
complex. The resulting chemical content of hops after drying de-
termines the characteristics of the beer brewed with them. As such,
prediction of specific chemical contents during drying, whether to keep
changes within a set limit, or to achieve a specific level of chemical
components after drying is possible. Upon analysis of chemical contents
during drying, three chemicals which uniformly increased or decreased
during drying were noted and chosen for algorithm development,
namely Myrcene, β-Ocimen and 1-octen-3-ol The same calibration to
test set split method was utilized for the analysis of these chemicals.
However, due to errors during chemical analysis, the overall dataset
was reduced from 134 measurements to 73 (49:24 split).

The best predictive models for all 3 of these chemicals were gained
using the normalized MUCVE-PLS models. Myrcene prediction on the
test set achieved r² values of 0.83, 0.73 and 0.64, respectively. The
RMSE errors can also be seen to be within acceptable margins, when
considering the variation of these chemical components.

The current predictive models are at a usable level of performance.
However, there is room for improvement. A number of future changes
will include altering the illumination utilized for hyperspectral imaging
to an illumination which includes a greater output within the blue-
green region of electromagnetic space (400–600 nm) in an attempt to
equalize it. This will give more information for both moisture content
and chromatic prediction models. However, due to the low SNR of the
V10E sensor in the range between 400 and 500 nm the noise to signal
ratio might still be high. Investigations into the prediction of other
chemical components would also be of great interest, in combination
with the illumination addition.

### Table 3

| Temperature | Bulk Weight of hops (kg) | R² adj | RMSE | AlCc | P Value | % Likelihood |
|-------------|--------------------------|--------|------|------|---------|--------------|
| 65 °C       | 12                       | 0.987  | 0.987 | 0.035 | 0.035   |              |
|             | 20                       | 0.997  | 0.997 | 0.015 | 0.018   |              |
|             | 40                       | 0.998  | 0.968 | 0.012 | 0.065   |              |
| 70 °C       | 20                       | 0.972  | 0.972 | 0.053 | 0.054   |              |

and the product temperature for the dry condition might be due to
environmental conditions (e.g. incoming air humidity changes) and
differences in size distribution in comparison to the other tests. How-
ever, in this trial the data between the two sensors is also clearly cor-
related.

### 3.4. Hyperspectral prediction of water content, chromaticity and aromatic components

The first stage of results is that of the retrieved average spectra for a
given batch of hops during the drying process. Fig. 9 illustrates the
variation in normalized reflectance for the 12 kg hop batch dried at
65 °C with an air speed of 0.35 m/s.

Fig. 9a illustrates that the most usable wavelengths were in the
region from 500 to 1010 nm. The trials were conducted under quasi-
production conditions using halogen lights which have a low irradiance
below 500 nm and as can be seen from Fig. 9a the utilization of this
region would introduce a significant level of noise to a small data set
and be detrimental to model performance. The low light emission issue
is further enhanced by the lower SNR of the V10E sensor in this range.
It can also be noted that the retrieved reflectance information is greater
above 700 nm than in the region between 500 nm and 700 nm. With
this is mind, the PLSR analysis was undertaken in the 500–1010 nm
range. The green peak present at roughly 550 nm (Fig. 9a), known as
the "green gap" (Gundlach et al., 2009) between the absorption peaks
of chlorophyll a and b at 430 and 664 nm and 460 and 647 nm
respectively, is reduced in size as drying occurs. This indicates a change
in anthocyanin concentration, a sub-group of flavonoids, which have an
absorption peak in the region of 530 nm (Kim et al., 2008). Also of great
interest is how the peaks and troughs present in the 700–1010 nm
region are removed as drying occurs. This development might be due to
the change in concentration of other polyphenolic compounds. These
include flavanols, flavan-3-ols, phenolic carboxylic acids and others as
prenylflavonoids and have both a hydroxyl group (O–H) and an
 aromatic hydrocarbon (C–H). The changes could thus be due to the third
overtone of the third overtone of OH and the fourth overtone of CH in
the region of 740 and 760 nm (Amadio et al., 2017; Clement et al.,
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However, the consistent RMSE performance between the calibration
and test sets for the reduced wavelength set shows the importance of
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length set PLS models, shown in Table 2, does, however, point to a
### 3.6 General discussion

Based on the extensive tests performed in the modified pilot plant system which was equipped with an array of several environmental and optical sensors, it was possible to gain a deeper understanding of dynamic changes in hop cones during convective drying. Impacts of varying process temperature, specific air mass flow, harvesting conditions, bulk density and size distribution were observed on both drying behavior and quality changes for hops. In terms of drying behavior and color retention, best results were achieved for bulk harvested on a dry day and the lowest bulk weights which in this case translates to the highest specific air throughput. These results are in line with previous research for hops (Rybka et al., 2017, 2019; LfL, 2019).

Due to the distinctive physical characteristics of hops, i.e. leaves and the spindle, the final moisture content varies different parts of the cone. Similar results have been reported for other aromatic and medicinal plants such as Lemon Balm (Cuervo-Andrade, 2011).

Bitter components after drying were not impacted by the drying conditions, bulk weights and weather conditions which is in line with the research of Forster and Beck (1985) who showed that losses during drying are very low. However, Zeisig (1970) stated that an increase in temperature from 55 °C to 65 °C leads to an increase in losses which could not be confirmed by this research as the levels stayed constant.

Hop Storage Index was also not impacted by the drying. Again, these results are somewhat in disagreement with previous literature (Zeisig, 1970; Doe and Menary, 1979). This might be due to the further development of hop drying systems which allow for gentler drying since these studies were conducted, as well as the difference in variety and origin of the raw material (LfL, 2019; Lutz et al., 2016; Mejzr and Hanousek, 2007; Münterer, 2017a, b).

HSI was successfully implemented to detect both time series moisture content and chromaticity in a real, scaled up, drying process. No effect of temperature, air humidity and shrinkage on the model performance were found in this study despite clear evidence from literature towards the contrary (Yao et al., 2013; Romano et al., 2011). This, amongst others, might be due to the low temperature range (ΔT = 5 °C) in which the investigations were conducted. These aspects need to be further investigated in future studies. Due to problems with the freeze-drying system it was not possible to investigate time series changes in aromatic components. However, it was possible to principally build sound models for the prediction of the aromatic component contents in question. For all investigations conducted models for reduced wavelength sets performed similar to the full wavelength set.

Nevertheless, as demonstrated by Kamruzzaman et al. (2016) and Su et al. (2020) non-linear methods might show more accurate and robust performance than linear approaches such as PLSR in food quality analysis. Moreover, recent studies have demonstrated that in the development of HSI algorithms it is imperative to statistically compare the performance of the developed algorithm with the actual performance of the comparison method (Shrestha et al., 2020). Thus, future work needs to pay close attention to finding the most appropriate modelling approaches including locally weighted partial least square regression (LWPLSR) (Su and Sun, 2017; Su et al., 2018) and deep convolutional neural networks (CNN). Furthermore, the accuracy of the underlying methods for chemometric correlation and possible bias caused by temperature differences within the physical system of HSI or resulting multi spectral systems in which they are integrated need to be verified.

### 4. Conclusions

The present study is a proof of concept of integration of non-invasive visual sensors in drying systems beyond the laboratory scale. By combination of the information gathered through a multitude of
different methods (sensor and data fusion), it was possible to assess the dynamic impacts of drying conditions on hop quality in an integrated way.

The results of the research undertaken show that, besides bulk weight and temperature, harvesting conditions and specific air mass flow have a significant influence on both the drying time and the color changes of hops during drying at identical conditions. Thus, in practice, it is important to find the optimum compromise between process

| Pre-processing | Metric/ Chemical | Calibration | Test | Wavelengths |
|----------------|-----------------|-------------|------|-------------|
|                | R²              | RMSE        | R²   | RMSE        |            |
| Norm.          | M.C.            | 0.96 0.23   | 0.95 | 0.24        | [658,664,839,901,920,1010] |
|                | CIELAB a*       | 0.81 3.25   | 0.81 | 2.79        | [620,665,902,912,1010] |
|                | CIELAB b*       | 0.74 2.23   | 0.63 | 1.90        | [504,522,588,827,865,901,920,1010] |
|                | Myrcene         | 0.89 2.27 × 10⁸ | 0.83 | 3.83 × 10⁷ | [505,554,575,605,690,833,931,941,946,971,1010] |
|                | β-Ocimene       | 0.76 4.12 × 10⁸ | 0.73 | 4.61 × 10⁷ | [507,557,651,659,920,946,951,1010] |
|                | 1-octen-3-ol    | 0.67 5.78 × 10⁸ | 0.64 | 5.24 × 10⁷ | [517,536,575,580,659,1010] |
| SNV            | M.C.            | 0.92 0.33   | 0.93 | 0.29        | [611,703,902,1010] |
|                | CIELAB a*       | 0.87 2.59   | 0.55 | 4.18        | [507,574,589,605,634,695] |
|                | CIELAB b*       | 0.59 2.60   | 0.48 | 2.73        | [569,758,769,817,925,1010] |
|                | Myrcene         | 0.82 3.33 × 10⁶ | 0.82 | 4.84 × 10⁶ | [536,565,592,897,982,1010] |
|                | β-Ocimene       | 0.79 3.92 × 10⁶ | 0.66 | 6.68 × 10⁶ | [533,583,597,659,684,1010] |
|                | 1-octen-3-ol    | 0.74 4.18 × 10⁶ | 0.63 | 6.96 × 10⁶ | [546,594,651,670,695,700,719,734,819,910] |
settings and scheduling on the one hand, and acceptable color changes on the other. The contents of α and β acid, as well as the Hop Storage Index, were not impacted by the drying conditions and, thus, can be seen as non-critical parameters in terms of process optimization.

The presented work illustrates the feasibility of moisture content and chromatographic information prediction during hop drying using a novel dryer and imaging system. The developed models have shown an acceptable level of performance, with future improvements likely to enhance this further. The design and combination of the hyperspectral and RGB imaging systems within the hop dryer are also noted to be novel and have shown a new way hops can be monitored, in real time, during drying. Further information fusion with process data helped to create a deeper understanding of the underlying processes within the hop cones.

Further work needs to include the deeper investigation of the losses of aromatic components which are highly volatile and, thus, susceptible to losses during drying. In addition, the characteristics of the drying curves need to be further investigated, and the interactions between the leaves and the spindle modelled. Different storage times pre-drying need to be evaluated for the determination of their impact on the drying leaves and the spindle modelled. Drying losses during drying. In addition, the characteristics of the drying of aromatic components which are highly volatile and, thus, susceptible to losses during drying. Further information fusion with process data helped to create a deeper understanding of the underlying processes within the hop cones.

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