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73023 Victoria Island, Lagos, Nigeria
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Full Length Research Paper

Malt roasting quality control by mid-infrared spectroscopy

Deborah Herdt[1*], Tobias Teumer[1], Sindhu Nair Balan[1], Nur Adibah Kamarulzman[1], Thomas Kunz[2], Sarah Kühnemuth[2] Frank-Jürgen Methner[2] and Matthias Rädle[1]

[1]Center for Mass Spectrometry and Optical Spectroscopy, Mannheim University of Applied Sciences, Mannheim, Germany.
[2]Institute of Food Technology and Food Chemistry, Technische Universität Berlin, Berlin, Germany.

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In the presented investigation, the chemical composition of malt during roasting is estimated using diffuse reflectance mid-infrared fourier transform (DRIFT-MIR) spectroscopy and multiple linear regressions. Accordingly, the corresponding test setup is presented and evaluated. A total number of sixty-five stop roasting, having temperature range from 140 to 220ºC, and one unroasted sample of 1500 g Avalon malt are performed in an eddy current roaster. Roasted and unroasted malt samples are milled and then analysed. Additionally, analytical standard reference methods are performed for colour, spectral tristimulus L*a*b* - values, colour difference (ΔE), iron-content, quantitative radical generation and the formation of specific intermediates, such as 5-(hydroxymethyl) furfural (HMF) as well as 3-deoxy-hexosulose (3-DH) and end products of Maillard reaction on all sixty-six samples. Multiple linear regression models were used to predict analysed references based on mid-infrared data, modified with spectral pre-processing for better prediction performance. The obtained results indicate that DRIFT-MIR spectrometry, combined with pre-processing and selection of evaluated wave number areas, is a useful analytical tool for the measurement of quality attributes of malt and therefore, shows potential for application in quality and process control.

Key words: Malt roasting, mid-infrared, optical spectroscopy, process control, EBC, L*a*b*, ΔE, quality control.

INTRODUCTION

Roasting of malt or barley is a crucial step of roast-, caramel- and malt production which defines physical, structural and chemical properties (Yahya et al., 2014). Several compounds like polyphenols, protein fraction, amino acids, etc. originating from barley, and Maillard reaction products (MRPs) (Carvalho et al., 2014), generated during malting, and are found in malt (Goupy et al., 1999; Shahidi and Ambigaipalan, 2015; Woffenden et al., 2001; Wunderlich et al., 2013). Chemical composition of ingredients as well as the physical properties of malt e.g. colour or flavour are altered through roasting processes (Coghe et al., 2006; Martins et al., 2000; Mohsin et al., 2018). The on-going automation in food industry and rising requirements on

*Corresponding author. E-mail: d.herdt@hs-mannheim.de.

quality control demand real time online analytic techniques. Near infrared (NIR) spectroscopy has been used in many studies before to monitor the quality of protein content in barley and malt (Fox et al., 2002; Ratcliffe and Panozzo, 1999; Sá and Palmer, 2006; Schmidt et al., 2009) as well as physicochemical changes (Contreras-Jiménez et al., 2019).

A study by Kljusurić et al. investigated the determination of the optimal process conditions for barley milk by combining NIR spectroscopy with measuring particle size and conductivity of samples (Gajdoš Kljusurić et al., 2015). Moreover, barley analysis by NIR (Czuchajowska et al., 1992) and mid infrared (MIR) spectroscopy (Cozzolino et al., 2013; Cozzolino et al., 2014) has shown to be useful for prediction models of malt quality. The possibility to control malt roasting and therefore, to define a stop criterion for targeted roast malt colour, is a challenging topic (Bamforth, 2017).

The malt colour shift is specified by MEBAK (Methodensammlung der Mitteleuropäischen Brautechnischen Analysenkommission) (Jacob, 2016) as EBC (European Brewery Convention) value (Miedaner, 2002). EBC value estimation is a method to determine the colour of wort and beer under defined conditions. Beer is decarboxylated; turbid samples are taken and afterwards; membrane is filtered. The EBC value is then determined by simply comparing the beer colour with a standardized scale. A previously established method, the Lovibond common technique, a visual method which is revised (Bishop 1950, 1966; Sharpe et al., 1992; Smedley, 1992). One possible error source of this method is that subjective perception of each person varies and, consequently, shows the differences in the colour values.

An alternative of the above-mentioned technique is spectrophotometric method in which subjective influences of the human eye are eliminated. This method is used in this work as a reference. The measurement is performed in a 10 mm cuvette at a wavelength of 430 nm (Farber and Barth, 2019; Miedaner, 2002). The sample is diluted to access absorbance in the linear range of the visible spectrophotometer. However, the accuracy of this method is quite low (Hans, 2009).

In addition to the EBC values, there is also the SRM (Standard Reference Method) (Miedaner, 2002) value, which can be converted into the EBC value using a factor of 1.97. Another measuring concept is considering different colours of varieties of beer, a method put forth by the Commission Internationale d'Eclairage. This technique comprises L*, a* and b* values and is called tristimulus CIE Lab colour (Bamforth, 2009; Mallet, 2014). This is used as a reference method to determine the change of colour during the roasting process. This study is conducted to identify optimal roasting temperatures with reference methods. The L*a*b* colour system is an international standard developed in 1976. By mathematical conversion, seemingly indiscernible colour differences when observed by the human eye can be expressed as differing measured values of approximately the same magnitude. The tristimulus colour system comprises of three dimensional colour space where L* constitutes the ordinate and represents the brightness (L* from luminance, Figure 1), which ranges from black (0) to white (100). The abscissa ranges from green (negative a* values) to red (positive a* values) and the applicate ranges from blue (negative b* values) to yellow (positive b* values). The both chromatic axes range from -120 to 120. ∆L*, ∆a* and ∆b* values describe the colour differences between one reference standard value ($L^*_{standard}$, $a^*_{standard}$, $b^*_{standard}$) and a sample value (L^*_{sample}, a^*_{sample}, b^*_{sample}). Based on these values the colour difference (∆E) is calculated with Equation (1) which describes the linear distance of the sample and the standard in the three dimensional colour system. (Ohta and Robertson, 2005; Otterstätter, 1999) Therefore, the delta E value can be used to define the approved colour difference in processes (González-Manzano et al., 2008; Pathare et al., 2013).

Using L*a*b* values colour difference, ∆E can be calculated by using the following equations (Ohta and Robertson, 2005; Otterstätter, 1999)

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

$$\Delta L^* = L^*_{sample} - L^*_{standard} \quad (2)$$

$$\Delta a^* = a^*_{sample} - a^*_{standard} \quad (3)$$

$$\Delta b^* = b^*_{sample} - b^*_{standard} \quad (4)$$

Likewise, other industries already use colour tolerances for ΔE values. For example, the evaluation of paper white (∆E = 3), which is stated in ISO 12647-7, is carried out with a spectrophotometer (Homann, 2010). The automotive industry also implemented a DIN (DIN 617-2) using 3 or 5 angle instrumentation, which is used by Audi for automatization of a process (Streitberger and Dössel, 2008). ∆E < 0.2 are not visible, 0.2 < ∆E < 0.5 are extremely poor visible, 0.5 < ∆E < 1.5 are poor visible, 1.5 < ∆E < 3.0 are visible and 3.0 < ∆E < 6.0 are strongly visible (Hellerich et al., 2010). Gierling, 2001 stated that a colour difference (∆E) below 1.0 is just visible to a professional, ∆E below 2.5 is not visible to a layperson and above 2.5 it is visible to a layperson. The purpose of this investigation is to compare standard methods for quality assertion with optical measurements of spectral properties in order to find out if this can be useful for further developments of at-line or in-line measurements to ensure high quality in the malt roasting process. The non-enzymatic browning, which is quantified with these methods, is dependent on the concentration of brown pigments in malt produced during pyrolysis of sugar (caramelization) as well as the reaction of the amino

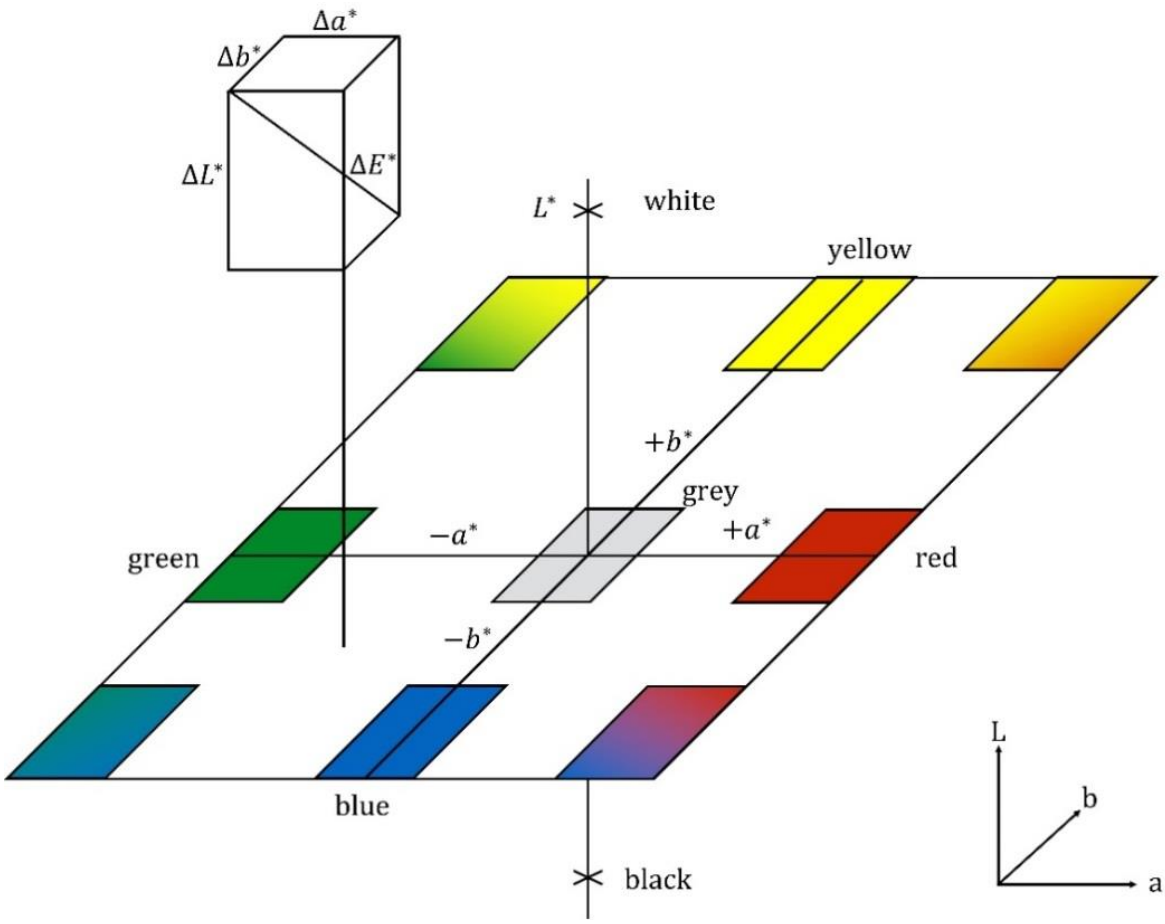

Figure 1. CIE- L*a*b* colour system: L* for the lightness from black (0) to white (100), a* from green (−) to red (+), b* from blue (−) to yellow (+) and quantifiable colour difference ΔE.
Source: Otterstätter (1999).

group of a free amino acid and the carbonyl group of a reducing sugar (Maillard reaction). These changes are reflected in altering chemical compositions and therefore varying vibrational modes. The goal is to investigate those changes in order to determine optimal roasting parameters. The Maillard reaction is a non-enzymatic browning reaction, which increases with temperatures (Labuza, 2005; Nie et al., 2013; O'Brien et al., op. 1998) due to increase in reactivity between the sugar and the amino group at higher temperature (Martins et al., 2000). By splitting off hydrocarbons, amino acids combine with reducing sugars to form a Schiff base. Some of these products (reductones, melanoidins) have a pro- or antioxidant effect and thus influence the oxidative stability of food (Cortés et al., 2010; Kanzler et al., 2017).

The formation of Maillard reaction products is influenced by temperature (Mohsin et al., 2018), time (Ćosović et al., 2010), water content (Faist et al., 2002; Yahya et al., 2014), pH-value (Kim and Lee, 2008; Kwak et al., 2005) and concentration of reaction partners (Spieleder, 2007; van Boekel, 2006). MRPs consist of aldehydes, acryl amides, dicarbonyls, ketones, heterocyclic amines and other compounds which are responsible for malt flavour and colour (Bravo et al., 2002; Cortés et al., 2010; Kanzler et al., 2017; Wang et al., 2011). An important product class is melanoidins, which are formed at the end of the Maillard reaction (Carvalho et al., 2014; Mohsin et al., 2018; Spieleder, 2007; van Boekel, 2006) and are mainly responsible for beer colour. Melanoidins with higher molecular weight are only formed at high temperatures. Melanoidin structures are largely unknown, with only a few being proposed. Hitherto, identification and characterisation of high-molecular, colour-intensive melanoidins are very challenging (Narziß et al., 2009). Additionally, another MRP 5-(hydroxymethyl) furfural is formed from hexose dehydration (Bertrand et al., 2018; Nursten, 2005). In this study, HMF is used as reference value for the intermediate stage of the Maillard reaction.

The other reaction involved in developing colour is sugar caramelization, which is the controlled

decomposition of sugars by pyrolysis. The process breaks down sugar molecules, evaporates the water and converts the remaining atoms into new flavours. In contrast to the Maillard reaction, here, no amino acid compounds are required. Moreover, it takes place at a higher temperature than the Maillard reaction, e.g. 110 for simple fructose and 180ºC for maltose. Caramelization of sugars produces both brown-coloured products with a typical caramel aroma and volatile aroma-active substances. In principle, the reactions correspond to those described in the Maillard reaction, except that the 3-deoxyosone is formed directly from the precursor hexose via 1,2-enolisation (Shahidi and Ambigaipalan, 2015).

Again, HMF is formed from hexoses and 2-furfural from pentoses (Kroh, 1994; van Boekel, 2006). HMF and furfural are intermediates formed during Maillard reaction due to 1,2 enolization (Martins et al., 2000). Additionally, acetic acid and vicinal diketones (diacetyl and 2,3-pentandione) formed after retro-aldolization are recognised as MRPs in malt (Coghe et al., 2006).

Furthermore, some of the compounds such as aliphatic alcohols, aldehydes, ketones, pyrroles, furans and pyrazines are identified in malted barley by gas chromatography (Beal and Mottram, 1994). Among the highlighted compounds, it is concluded that 3-methylbutanal and 2-methylbutanal increase dramatically in malt during roasting. Another study analysed fifteen Maillard products related to flavour development by roasting three different kinds of malt (Yahya et al., 2014). (Yahya et al., 2014) focused on the trend of maltol concentration in malt and the correlation with other compounds such as 2-furaldehyde, methyl-pyrazine, Isomaltol,2-furanmethanol, 2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one. Gupta et al., 2010 emphasized notable traces of β-Glucan, protein, fibers, arabinoxylans in by-products left after separating wort during the brewing process. Hydrolysis of starch carried out by malt enzymes during the malting process is also mentioned. The resulting fermentable sugars or simple sugars from this process are glucose, sucrose, fructose, maltose and dextrin (Gupta et al., 2010). Research on xylose content changes in malt is also conducted, concluding that xylose content in malt varies in a noticeable way (MacLeod et al., 1953). In this research, spectral changes in mid-infrared regions are investigated regarding biochemical, as well as colour changes during the roasting of malt. A correlation between reference values and spectral data were described by multi linear regressions.

MATERIALS AND METHODS

Kiln malt of the Avalon variety (provided by Palatia Malz GmbH, Wallertheim, Germany) was roasted in an eddy current laboratory roaster. Roasting test series were performed at different temperatures and roasting times. Roasting was stopped in 60 s intervals with a maximum exposure time of 780 s. For each specified roasting temperature (140, 160, 180, 200, 220ºC), 12 roasting test series were carried out. Roasted malt samples were then milled manually using a coffee grinder in order to get a small particle size distribution to measure as closely as possible to the process.

Additionally, the used diffuse reflection infrared Fourier transform (DRIFT) spectroscopy method required the utilization of small sample volumes. The malt samples were therefore analysed with DRIFT spectroscopy as grist. The roasted milled samples (grist) are displayed in Figure 2, starting in the left upper corner with 120 60 s and ending with 240ºC 780 s in the right lower corner. Each row showed a specific roasting temperature and the columns represent the roasting times 60 s, 120 s, 180 s, 240 s, 300 s, 360 s, 420 s, 480 s, 540 s, 600 s, 660 s, 720 s and 780 s.

Standard wort analysis

The basic chemical analysis for the characterisation of roasted malt samples was carried out by the Chair of Brewing and Bewerage Technology, Institute of Food Technology and Food Chemistry, Technical University of Berlin in accordance with the regulations of the Central European Brewing Analysis Commission (MEBAK) (Methner, 2018). Standardized and special reference methods were used in order to verify the roasting status. Malt was coarsely milled with an analytical mill (Brühler- MIAG A10, IKA Labortechnik), 75 g (90 roasted malt and 10% Pilsner malt) grist was mixed with 300ml double distilled water (55ºC) stirring constantly in a beaker. The grist was malted in a mash bath according to the following program: 5 min at 55, 5 min at 60, 20 min at 62, 25 min at 65, 20 min at 72 and subsequent heating-up to 78ºC. After reaching 78ºC, the beaker was taken out of the bath and rinsed with double distilled water. The total mass was filled with water to 450 g and subsequently filtered with a fluted filter (Whatmann Filter 597/2). 100 ml of the eluat was set aside as a sample before wort boiling for reference analysis regarding colour values (L*a*b* before wort boiling). The remaining 300 ml was cooked in the beaker under reflux for one hour, filtered with a fluted filter (Whatmann Filter 597/2) and cooled down before EBC colour of malt extract (MEBAK R-205.07.110 [2016-03] (Methner, 2018) 2.13.2. (Miedaner, 2002) and then L*a*b* was measured. Until further analysis, samples were kept frozen in storage. Solid samples were used for inductively coupled plasma optical emission spectroscopy (ICP-OES). High-performance liquid chromatography (HPLC) and Electron Spin Resonance (ESR) spectroscopy were carried out on the resulting eluat sample after boiling wort. Reduction capacity (Red. Cap. cf. MEBAK 2.16.1 (Miedaner, 2002)) and radical levels (T_{600} and ESR value cf. MEBAK 2.15.3 (Kunz et al., 2013; Methner et al., 2007; Miedaner, 2002; Uchida and Ono, 1996)) were determined according to MEBAK. The organic radical content of roasted malt was quantified using an optimized ESR spectroscopy method, patented (Kaneda et al., 2005) and described in a paper by (Takoi et al., 2003). The ESR measurement was optimized by the Technical University of Berlin, considering a method by (Cortés et al. 2010). Early Intermediates from Maillard reaction, for example α-dicarbonyls, such as 3-deoxy-hexosulose as well as HMF, were measured by High-Performance Liquid Chromatography with Diode-Array Detection (HPLC-DAD).

Determination of α-Dicarbonyl compounds via HPLC-DAD (Kanzler et al., 2017)

The quantification of α-Dicarbonyls (e.g. 3-DH) was carried out by high-performance liquid chromatography with diode-array detection after derivatization with *ortho*-Phenylendiamine (OPD). 5 ml of liquid

Figure 2. Milled roasted malt samples.

Table 1. HPLC DAD gradient of α-Dicarbonyl compounds.

Time (min)	Methanol (%)
0	20
5	20
35	60
40	60
45	20
55	20

sample was mixed with 1 ml 0.05 M OPD solution and derivatized at room temperature for 24 h. The solution was stored at – 18ºC until measurement. Prior to analysis, samples were filtered with a syringe filter (Nylon; 0.45 µM). A reverse phase column from Knauer (60-5 phenyl) was used for separation. The solvent gradient (methanol and water) was listed in Table 1. The flow rate for identifying α-Dicarbonyls was 0.5 ml/min. Calibration was performed with a standard. 40 µl standard mix or sample was injected. The temperature of the column oven was set to 35ºC. Detection and quantification was performed at 318 nm.

Determination of HMF via HPLC-DAD (Kanzler et al., 2017)

HMF served as indicator or marker substance for the formation of particular heterocyclic intermediates of the Maillard reaction. HMF was determined using an HPLC-DAD method described below (Kanzler et al., 2017). A reverse phase column from Knauer (60-5 phenyl) was used for separation. The solvent gradient consisted of methanol and phosphate buffer as listed in Table 2 at a flow rate of 0.5 ml/min. Calibration was performed with five different concentrations of a HMF standard. 20 µl standard mix or sample was injected. The temperature of the column oven was set to 35ºC. Detection and quantification was performed at 285 nm.

Determination of Iron entry via ICP-OES

The individual caused iron entry, dependent on roast status of grain samples, was measured using an ICP-OES system iCAP 6200 equipped with a CID 86 detector and an auto sampler from Thermo Scientific. Solid samples were digested beforehand by microwave. 0.25 g of solid samples was decomposed with 5 mL HNO_3 and 2 mL H_2O_2 and then placed in a Teflon vessel. The mixture was incubated for 1 h at room temperature and subsequently placed in a microwave oven for 1 h at 160ºC. Afterwards the sample was cooled for 30 min and filtered. All vessels and the filter were rinsed with demineralised water; further demineralised water was added to the solution until a total volume of 25 ml was reached for the following analyses via ICP-OES. Liquid samples were generated as described for the standard wort analyses. Subsequently, the defrosted wort samples were diluted with a dilution factor of 10 or 20, depending on wort colour, for the analyses via ICP-OE Avio 200 system equipped with a CCD detector and an autosampler. The iron concentration was determined by external calibration using ICP-OES with argon as carrier gas and emission lined at 238.2 nm and 259.9 nm.

Tristimulus colour measurement of L*a*b* values

The measurement of L*a*b* values was conducted using a

Table 2. HPLC DAD gradient of HMF.

Time (min)	Methanol (%)
0	5
5	5
15	20
20	20
25	95
35	95
40	5
45	5

photometer (Konica Minolta VC 5, illuminant D65 (DIN 6173-2) observer angle of 10º). The liquid samples (wort before and after boiling) were membrane filtered (25 mm Syringe filter, w/0.45 µm cellulose acetate). After required liquid calibration (0 and 100 % calibration) the samples were measured in 10 mm plastic cuvettes (triple determination). Grain kernels or milled grist were transferred into petri dishes for L*a*b* value determination. Due to the inhomogeneous solid samples (grain and grist), a fivefold determination with different rotation positioning of the petri dishes was performed.

Fourier-transformed infrared spectroscopy (FTIR) Spectroscopy

FTIR monitored the interaction of functional groups in chemicals molecules. The spectra were recorded with the FTIR spectrometer ALPHA-R from Bruker (Bruker Optics GmbH, Ettlingen, Germany) using the DRIFT (Diffuse Reflectant Infrared Fourier Transformation) module. Freshly milled malt was used for each measurement and the samples were scanned three times and the average ATR-FTIR spectrum was used for further analysis. The spectra, with 4 cm^{-1} resolution in the range from 4000 to 400 cm^{-1}, were recorded on OPUS software version 7.0 (Bruker Optics) and each sample was obtained by calculating the average of 24 scans. Gold was used as reference background spectra. The DRIFT sample compartment was cleaned before each sample was scanned. Freshly milled malt was used for each sample measurement. The resulting absorption spectra were cut off below 800 cm^{-1} and above 3700 cm^{-1}. Baseline correction (concave elastic band method, using 25 iterations and 30 baseline points) and smoothing (17 smoothing points) were performed, resulting in the spectra used for evaluation. Due to the complex biochemical matrix of the sample and therefore overlaying spectral changes, chemical standards were not measured. The spectra were evaluated by identifying peaks (b-f) and calculating the peak areas to the integral border.

Statistical Analysis

Regression analysis was a statistical evaluation method with the objective of describing a context by a function. The objective was to find a dependency between the dependent variable y and the co-variable x. Target quantity of the covariates could not be described exactly by the function, but were rather affected by disturbances. The target variable became a random variable, because its size depended on the distribution of the covariates. This meant that, without exception, the mean value of the covariates could be used to deduce the target variable. The distribution could not be predicted, therefore, the average was used for the calculation. The target variable could be described by a linear, quadratic or exponential function. The most frequently used regression model was linear and described by the following standard Equation (5):

$$y = \beta_0 + \beta_1 \cdot x_1 + \ldots + \beta_n \cdot x_n + \varepsilon_i \quad (5)$$

where ε_i is an error variable and β_n (with n=0, 1, 2,…) are unknown estimated parameters. For multiple linear regressions, multiple independent variables were used. A benchmark for linear relationship or the normal distribution was the coefficient of determination R^2. The coefficient of determination is expressed by the following Equation (6) (Fahrmeir et al., 2009):

$$R^2 = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \quad (6)$$

$\hat{y} = \text{arithmetic mean of calculated values}$
$\bar{y} = \text{arithmetic mean of reference values}$
$y_i = \text{reference values}$

The number of degrees of freedom (df) was the number of values which remain in the final calculation of a varying statistic and it was calculated with Equation (7) for a linear regression (Kessler, 2005):

$$df = n - 2 \quad (7)$$

$n = \text{number of reference values}$

Additional coefficients of regression reduced the degree of freedom by one (Kessler, 2005). The degrees of freedom were used to calculate the residual standard error (RSE), which was the positive square root of the sum of the squared residuals divided by the degrees of freedom (Kessler, 2005), thus it was calculated by:

$$RSE = \sqrt{\frac{1}{df}\sum_{i=1}^{n}(y_i - \hat{y}_i)^2} \quad (8)$$

The independent variables were the calculated values of the areas b, c, d, e and f for all roasted samples. The independent variables were both temperature dependent ($T_{i\ldots m}$ for temperature ranging from i to m) and time dependent ($t_{i\ldots n}$ for roasting time ranging from i to n). Target variable corresponds to the reference values (EBC, iron content, TMAX value, HMF, 3-DH, reduction and L*, a*, b* values). For each reference value, a multiple regression model was created according to Equation (9) (Groß, 2003).

$$Abs. = f(T,t) \Rightarrow A = \begin{bmatrix} T_1t_1 & T_1t_2 & \ldots & T_1t_n \\ T_2t_1 & T_2t_2 & \cdots & T_1t_2 \\ \vdots & \vdots & & \vdots \\ T_nt_1 & T_1t_2 & \ldots & T_mt_n \end{bmatrix} \quad (9)$$

The R software (version 3.5.1, 2.07.2018) was used for pre-processing. For evaluation, a partial least square regression (PLSR) model was used and a scatterplot, a prediction plot and the coefficient of determination were generated.

RESULTS AND DISCUSSION

The resulting processed spectra of the samples were evaluated, concerning the changing areas of vibrational

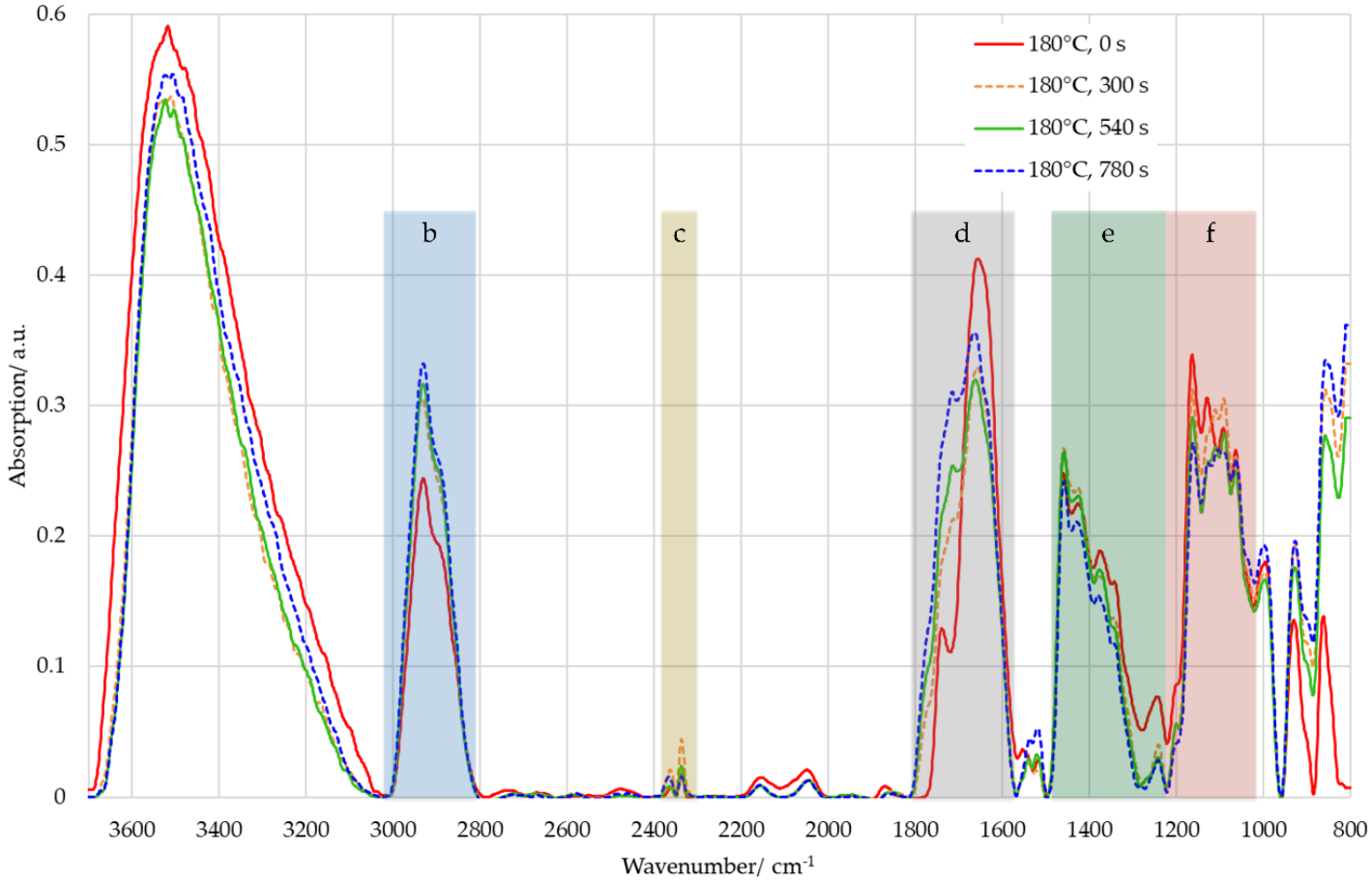

Figure 3. Processed spectra of roasted and unroasted malt with evaluated areas (variables) b-f.

changes during the roasting process. The first derivative was calculated in order to identify the spectral peak maxima, which represented relevant changes. As shown in Figure 3, the stretching vibration of water in between 3700- 3000 cm^{-1} changed due to the varying water concentration of the samples. However, the malt samples could have been influenced by the environment after milling and even during DRIFT measurement and therefore the range, where OH- absorption was visible, was not taken into account. The analysed peaks were in between 3027- 2787 cm^{-1} (b), 2362- 2307 cm^{-1} (c), 1807- 1567 cm^{-1} (d), 1500-1280 cm^{-1} (e) and 1218-962 cm^{-1} (f) (Figure 3). These ranges were used to calculate the areas underneath and then used as independent variables b, c, d, e and f for regression analysis.

The observed range for peak (b) was assigned to the stretching vibration of aliphatic carbon hydrogens (vC-H) at 2850- 2960 cm^{-1}, the stretching bond of amino groups (vN-H_3^+C-H) at 2600-3100 cm^{-1} and carboxyl acids at 2400-3300 cm^{-1} (Gunzler and Gremlich, 2003). Peak (c) was designated to the asymmetric stretching vibration of carbon dioxide (vCO_2 at 2349 cm^{-1}) (Groß, 2003) or aryl compounds (2309-2136 cm^{-1}) (Gunzler and Gremlich, 2003). In the region of 1807- 1567 cm^{-1} peak (d), the stretching vibration of carbonyl compounds (e.g. ketones, acids, esters, amides, etc.) as well as the deformation vibration of water (δH_2O) were located. Peak (e) was designated to the stretching vibration of aldehydes, deformation vibration of melanoidins or methyl groups. In the fingerprint region below 1500 cm^{-1} (peak f) each compound itself had a unique combination of peaks due to deformation vibrations generated by C-C and C-O interactions in ethers, alcohols, esters, etc. or stretching vibrations from C-N interactions from amines, amino acids or amides (Schmidt, 2000).

Table 3 lists the individual vibrations and the possible educts, intermediates or products which were influenced during roasting. In Figure 4, the results of standard wort analysis radical levels (ESR value cf. MEBAK 2.15.3 (Miedaner, 2002)) and the corresponding standard deviations were displayed as an example. The temperatures in between 140 to 180ºC showed a low ascent of organic radicals in contrast to the higher temperatures. The organic radical concentration

Table 3. Peak ranges and assigned vibrations and possible substances.

Peak label/ variable	Vibration
b	vC-H methyl group: 1-Deoxyosone and 3-Deoxyosone vC-H aromates (Gunzler and Gremlich, 2003); HMF (Nikolov and Yaylayan, 2011)
c	$v_{as}CO_2$ (2349) (Gunzler and Gremlich, 2003) or Aryl compounds (Gunzler and Gremlich, 2003)
d	vC=O and vC=N and vC=C Melanoidines (Cämmerer and Kroh, 1995; Rubinsztain et al., 1986) vOH carboxylic acid esters vC=O ketones vC=N carboxylic acid amides (Gunzler and Gremlich, 2003) δH_2O (Gunzler and Gremlich, 2003)
e	vC-C aldehydes: glucose Aryl-O-Ether (Gunzler and Gremlich, 2003) δOH melanoidins (Cämmerer and Kroh, 1995) δC-H methyl group: 1-Deoxyosone and 3-Deoxyosone (Cämmerer and Kroh, 1995; Ledl and Severin, 1978)
f	vC-O polysaccharides resulting from ether or carboxylic acids: aldoses (reducing sugar) or melanoidins (Cämmerer and Kroh, 1995) v_{as}C-C ketones (Gunzler and Gremlich, 2003) ethers (HMF -> aldehyd and ether) vN-H secundary amines (acrylamide and amino acids) or melanoidins (Cämmerer and Kroh, 1995) vN-H aromatic amines: pyridine (amadori product)

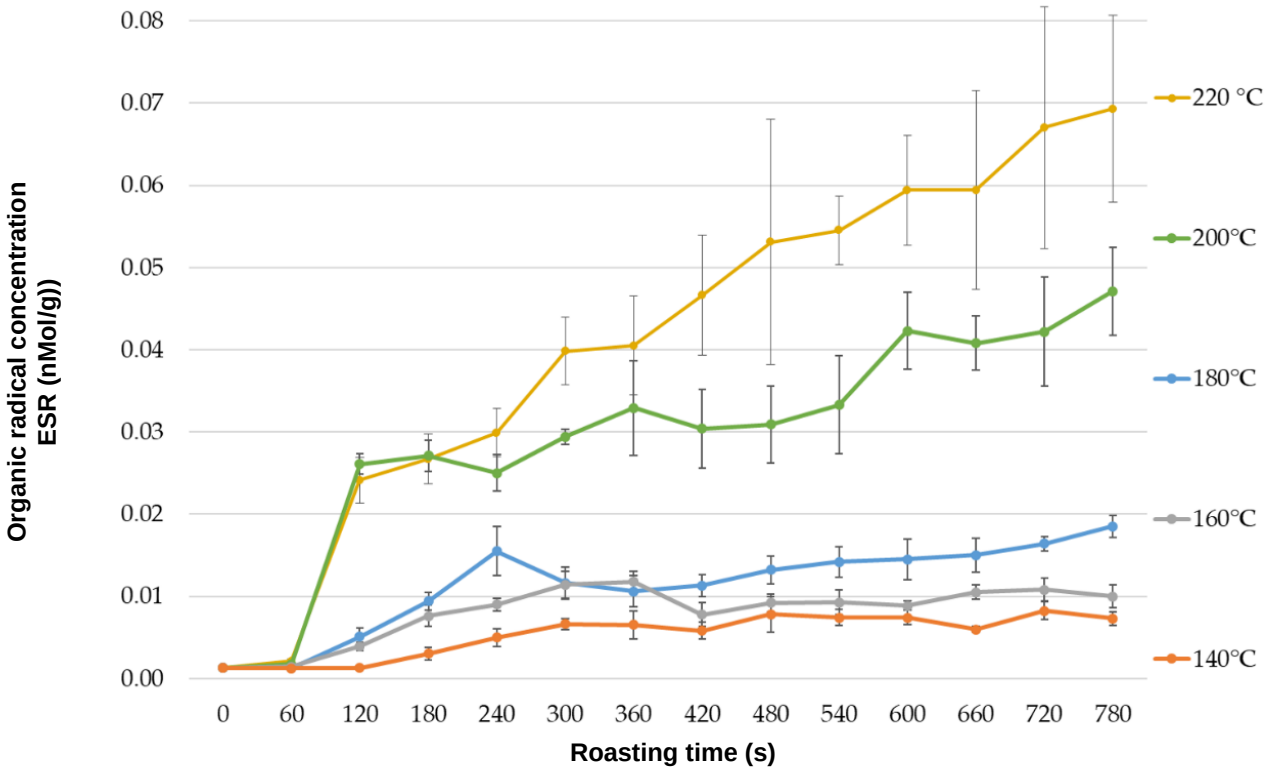

Figure 4. Organic radical concentration ESR over roasting time.

increased with roasting time and was highest for 220ºC.

Figure 5 exemplary displayed one resulting scatterplot matrix for the L* value of the grist (variable a), compared to the evaluated peaks (variable b- f). It was used to describe the correlation between the reference value (a) and variables b-f. The scatter plot matrix was used to

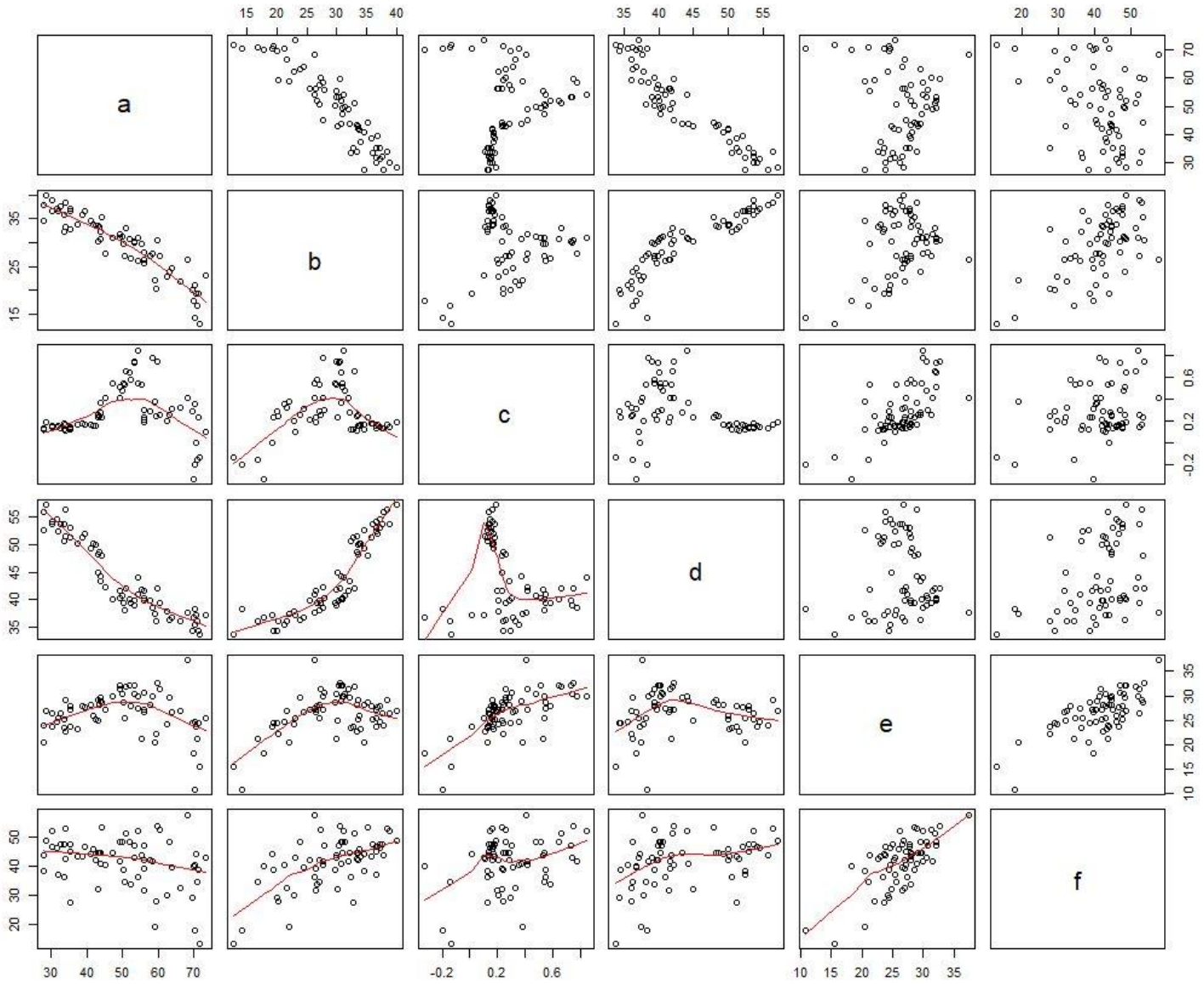

Figure 5. Scatter plot matrix for a= L*, grist and b-f= calculated peak areas.

select important variables and showed their relationship to each other. As shown in Figure 5, the L* value of grist (variable a) and the mid-infrared data in between 3027-2787 cm^{-1} (variable b) had a strong linear positive correlation. In contrast, variable (a) and (c) had no distinct linear relationship and showed a weak correlation as compared to variable (a) and (b).

Figure 6 (a) displayed the result of the multiple linear regression with the L*- values of the grist generated from the software. The prediction plot on the left side showed the predicted against the measured L*-values of grist. The linear regression was normally distributed, which could be seen on the right graphs (Figure 6 (b) – (e). The residuals vs fitted plot (Figure 6b) indicated that the residuals and the fitted values were uncorrelated, as they should be in a homoscedastic linear model with normally distributed errors. Most residuals were close to zero in contrast to the more extreme residuals which were far away from the rest. This indicated that the outliers were sample number 1 (unroasted), 19 (160ºC at 300s) and 34 (180ºC at 420 s). Those outliers were also visible in the normal Quantile-Quantile (Q-Q) plot and the scale location plot. In (Figure 6c) the standardized residuals were shown on the vertical axis and were compared to the theoretical quantiles in the Q-Q plot. The normal Q–Q plot compared the randomly, independent standard normal data to a standard normal population. As shown below, the data points essentially formed a straight line

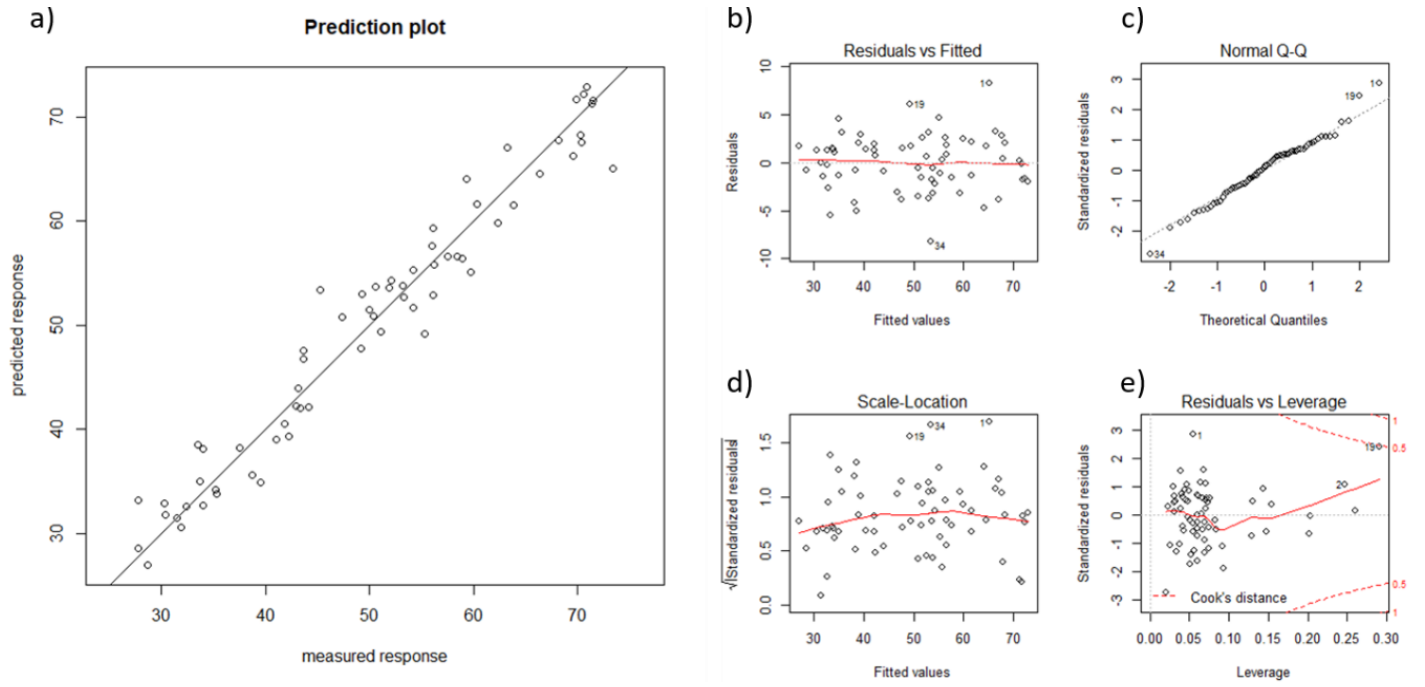

Figure 6. (a) Predicted vs. measured L* –values (grist), (b) residuals vs. fitted plot, c) normal QQ plot, (d) standardized residuals plot, (e) residuals vs Leverage plot with cook's distance.

which indicated that it was normally distributed. The Scale-Location plot (Figure 6d) showed that the residuals were spreaded equally along the predictor range of the L* grist variable. Since there was no any clear recognizable pattern of the measured L* grist values; the uniform variance was shown. The residuals vs leverage plot in (Figure 6e) showed that sample number 19 had a high influence on the regression line in contrast to sample number 1 and 2 (140°C at 60 s). Since the data sets were independent to each other and normally distributed, multiple linear regression was used. For further analysis and studies, the outliers, samples 1, 19 and 34, should not be considered.

In Figure 7, the calculated predicted data, on the basis of the independent variables b-f, was plotted against the measured reference values. On the left side in Figure 7, the prediction plot of the L*- value of grist was displayed and on the right side the prediction plot of the EBC value was shown. The measured L* values of grist ranged from 27.77 (beginning of roasting) to 73.37 (ending of roasting) and the coefficient of correlation was 0.953 with a standard deviation of 3.02. In contrast, the EBC values ranged from 30 to 780 and the coefficient of determination was 0.845 with a standard deviation of 78.19. The outliers in the EBC regression model, especially at the end of the roasting, could be seen at values above 580.

In Table 4, the resulting coefficients of determination (R^2) as well as the residual standard error (RSE) and the corresponding degrees of freedoms (df) were summarised. For the multiple linear regressions, all independent variables b-f were used to calculate the coefficients of determination. The results were compared to the linear regression models using variables (b), (f) separately as well as both.

Variable b showed a high correlation to referenced colour values and was, therefore, individually examined. In order to show the contribution of a second measured wavelength, variable f was taken as an example. The comparative low R^2, used together with variable (b), enhanced the model and additionally, proved that not all used variables had the same contribution to the model fit if taken into account. Due to the possible photometric measurement of the data, just the two variables (b) and (f) were taken as example. With measured variable (b) only, the coefficient of determination would not be robust for further implementing in roasting processes. The same applied for variable (f). In contrast, if both were included, significant coefficients of determination were calculated and therefore could be used in future developments of process automatization. However, the L*-, a*- and b*-values taken on their own did not give any information about the true colour. Therefore, the colour difference value ΔE was calculated for L*a*b* values of grain, grist, wort before and after boiling by using the unroasted sample as standard. The resulting margin of the values, R^2, df and RSE were summarized in Table 5. The ΔE values for grain varied in between 3.11 and 46.14 and

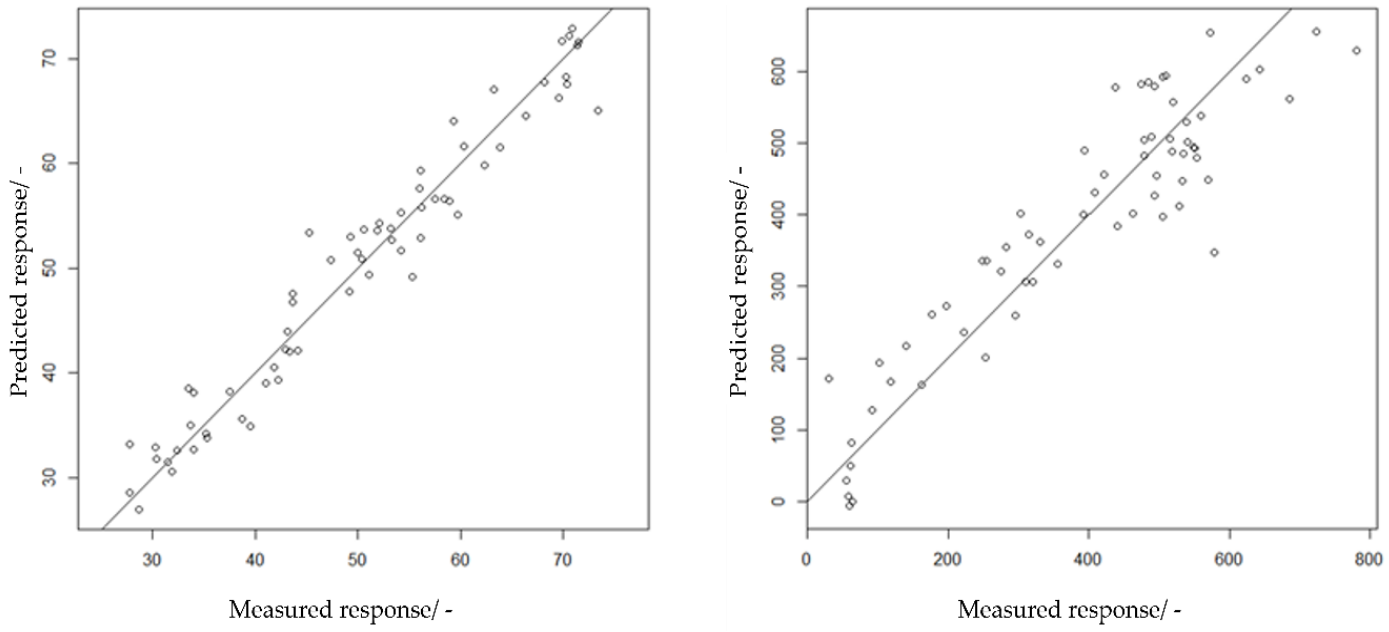

Figure 7. Comparison of prediction plot of (left) L value of grist and (right) EBC value.

Table 4. Calculated R^2 with varying dependent variable a (reference values) in the first column, covariables b-f (peak areas), designated RSE and df.

Variable a	Variables b, c, d, e and f			Variables b and f			Variable b			Variable f		
	df	R^2	RSE	df	R^2	RSE	df	R^2	RSE	df	R^2	RSE
EBC	59	0.845	78.190	62	0.835	78.800	63	0.766	92.990	63	0.117	180.700
Iron concentration	53	0.484	277.300	56	0.002	375.200	57	0.001	372.200	57	0.000	372.300
T_{600}	58	0.629	1.499	61	0.510	1.680	62	0.462	1.746	62	0.065	2.303
HMF	42	0.867	0.233	45	0.806	0.271	46	0.681	0.344	46	0.122	0.571
3-DH	42	0.728	630.300	45	0.693	646.900	46	0.597	733.100	46	0.058	1121.000
Red. Capacity	58	0.600	103.400	61	0.566	105.100	62	0.543	107.000	62	0.121	148.400
ESR	60	0.841	0.008	63	0.675	0.010	64	0.629	0.011	64	0.102	0.017
L*, grain	60	0.883	2.631	63	0.875	2.657	64	0.806	3.288	64	0.119	7.000
a*, grain	60	0.667	0.552	63	0.640	0.561	64	0.620	0.571	64	0.140	0.860
b*, grain	60	0.826	1.337	63	0.700	1.711	64	0.601	1.958	64	0.053	3.017
L*, grist	60	0.953	3.021	63	0.930	3.597	64	0.842	5.344	64	0.110	12.690
a*, grist	60	0.707	0.988	63	0.472	1.293	64	0.457	1.301	64	0.103	1.672
b*, grist	60	0.767	1.928	63	0.335	3.174	64	0.270	3.300	64	0.014	3.835
L*, wort before boiling	60	0.881	9.111	63	0.811	11.190	64	0.756	12.620	64	0.123	23.940
a*, wort before boiling	60	0.438	10.150	63	0.122	12.390	64	0.088	12.520	64	0.002	13.100
b*, wort before boiling	60	0.821	12.780	63	0.803	13.080	64	0.738	14.980	64	0.107	27.620
L*, wort after boiling	60	0.878	8.152	63	0.800	10.200	64	0.842	5.344	64	0.131	21.080
a*, wort after boiling	60	0.515	8.959	63	0.282	10.630	64	0.227	10.950	64	0.012	12.380
b*, wort after boiling	60	0.876	10.860	63	0.837	12.130	64	0.779	14.030	64	0.124	27.910

resulted in R^2 (b,c,d,e,f (ΔE, grist))=0.699 and a RSE of 1.927. The coefficient of determination for the colour difference of grist was 0.956 with a standard error of 2.722, which meant that there was a very strong correlation between the ΔE values and the analysed MIR data. The coefficient of determination of ΔE for wort

Table 5. Value margin of ΔE values and corresponding coefficients of determination by using variables b-f

Variable	df	R^2	RSE	Values margin of ΔE
Δ E, grain	59	0.699	1.927	3.11-46.14
Δ E, grist	59	0.956	2.722	27.07-42.26
Δ E, wort before boiling	59	0.917	6.743	20.53-100.32
Δ E, wort after boiling	59	0.910	8.055	19.7-106.09

Table 6. Coefficients of determination for EBC, HMF, L*, grist and Δ E, grist with added temperature (g) and time (h) as variables

Variable	Statistical analysis	EBC	HMF	L*, grist	Δ E, grist
g	df	63	46	64	63
	R^2	0.3202	0.4622	0.4836	0.4501
	RSE	158.500	0.4471	9.666	9.337
h	df	63	46	64	63
	R^2	0.4621	0.0750	0.4081	0.3967
	RSE	141.000	0.5863	10.350	9.780
b, c, d, e, f, g, h	df	57	40	58	57
	R^2	0.9068	0.9093	0.9741	0.9742
	RSE	61.690	0.197	2.273	2.127
b, g	df	62	45	63	62
	R^2	0.7661	0.7579	0.8568	0.8619
	RSE	93.720	0.303	5.130	4.717
b,h	df	62	45	63	62
	R^2	0.8365	0.6814	0.8764	0.8865
	RSE	78.360	0.348	4.766	4.277
b,g,h	df	61	44	62	61
	R^2	0.8590	0.8087	0.9335	0.9323
	RSE	73.350	0.273	3.524	3.329
b,f,g,h	df	60	43	61	62
	R^2	0.8818	0.8791	0.9655	0.8467
	RSE	67.730	0.219	2.558	4.969

before (R^2=0.917) and after boiling (R^2=0.910) were similar due to the sample treatment. The wort before boiling was cooked under reflux for one hour and filtered to obtain the wort after boiling, which was done to compare the sample (wort after boiling) as close as possible to the brewing process itself, though the colour of the sample did not change a lot during the second cooking.

Additionally, roasting temperature and time could be used for further stabilizing the model. In most roasting processes, time and temperature were already available data sets and if incorporated in the model, the coefficient of determination could be further improved. If the multi-linear regression model, using the existing variables b-f, was extended with variable (g) as temperature and variable (h) as time, the coefficients of determination for EBC values could be enhanced from R^2(b,c,d,e,f (EBC)) = 0.8451 to R^2(b,c,d,e,f,g,h (EBC)) = 0.9068 (Table 6). The coefficients of determination for HMF and L*, grist were increased from R^2(b,c,d,e,f (HMF))= 0.8761 and R^2(b,c,d,e,f (L*, grist)) = 0.9527 to R^2(b,c,d,e,f,g,h (HMF)) = 0.9093 and R^2(b,c,d,e,f,g,h(L*,grist)) = 0.9741. A photometric measurement of two wavelengths, or in our case variable (b) and (f), could be combined for prediction models of the EBC value, Δ E values, HMF concentration or the L*- value of grist. By using the temperature (g), time (h), the ranges 3027- 2787 cm^{-1} (b) and 1219- 962 cm^{-1} (f) as variables, the calculated coefficients of determination were R^2(b,f,g,h (EBC)) = 0.8818, R^2(b,f,g,h (HMF)) = 0.8791, R^2(b,f,g,h (L*,grist)) = 0.9655 and R^2(b,f,g,h (ΔE,grist))= 0.8467.

The coefficients of determination for all five variables

varied in between R^2(b,c,d,e,f(a*,wort before)) = 0.438 for reference a*- value of the wort before boiling and R^2(b,c,d,e,f(L*,grist)) = 0.953 for reference L*- value of the grist. Some coefficients of determination showed a weak correlation between the independent variables and the reference values, like iron concentration (R^2 = 0.484), Tmax (R^2 = 0.629), 3-DH (R^2= 0.728) and the reduction capacity (R^2 = 0.600). Similarly, the coefficient of determination for a* of grain (R^2 = 0.667), grist (R^2= 0.707), wort before boiling (R^2= 0.438) and after boiling (R^2= 0.515) as well as the b* value of grist (R^2= 0.767) did not show a strong correlation.

In contrast, strong correlations were found for HMF R^2(b,c,d,e,f(HMF)) = 0.867, ESR R^2(b,c,d,e,f(ESR)) = 0.841 and L* R^2(b,c,d,e,f(L*,grist)) = 0.953. Considering the variables separately, the coefficients of determination for L*, grist were R^2 (b(L*, grist))= 0.842 and R^2(f(L*,grist))= 0.110. The correlation of the reference value and the variable (b) would mostly result in a weak correlation, except for the correlation between the L*-value of grain, grist and the wort before boiling. As shown in Table 4, range of R^2 for variable (f) was in between R^2 (f(a*, grain)=0.140 and R^2(f(Iron conc.))=0. Therefore, the variable (f) itself resulted in no correlation for all evaluated reference values. The results for variable (b) varied in between R^2 (b(iron conc.))= 0.001 and R^2 (b (L*, grist)) = 0.842. The coefficients of determination for L*- value of grist overall showed strong correlations. By using the EBC value, with only one variable for the multi linear regression, the resulting correlation was weak ranging from R^2 (f(EBC))= 0.117 to R^2(b(EBC))=0.766. However, taken both variables into account, a good correlation with R^2 (b), (f) (EBC))=0.835 could be achieved. The significance of the evaluated variables could be seen, if all variables were used to calculate the coefficient of determination. For the EBC value, the coefficient of determination was increased by 0.01 up to R^2 (b,c,d,e,f (EBC))= 0.845. Therefore, variables c, d and e all together did not show an impact on the correlation.

The correlation of the grinded samples (grist values) displayed the highest correlation by using the colour reference values L*a*b. Overall, most colour reference values showed a good coefficient of determination utilizing all variables: EBC (R^2= 0.845), L*, grain (R^2= 0.883), b*, grain (R^2= 0.826), L*, grist (R^2= 0.953), L*, wort before boiling (R^2= 0.881), b*, wort before boiling (R^2= 0.821), L*, wort after (R^2= 0.878), b*, wort after (R^2= 0.876).

As displayed in Figure 6, the data sets 1, 19 and 34 were outliers. If left out, the resulting coefficient of determination for the L-value of grist R^2 (b,c,d,e,f) increased from 0.953 to 0.969. Due to the roasting process test, the samples were roasted beyond their optimal colour and flavour and therefore, the model could be further optimized by using only pertinent values before and right after the stop of the roasting. This could be further used as a basis for a photometric measurement in malt roasting. However, the methodology presented must be an at-line method only at this stage, due to the used MIR spectroscopy. The application as inline measurement would require a system that can cope with elevated temperatures, which is not given with most MIR measurements. Considering the reference values, the correlation of the grinded samples (grist values) displayed the highest correlation by using the colour reference values L*a*b* or ΔE. This was due to spectral data, which was used as basis to calculate the evaluated areas. These data were obtained from the FTIR measurement of grist as well as the reference values. If the sample was further processed, the evaluated reference values still resulted in comparably high correlations for the L*- values. The dependent variable a (e.g. HMF, ESR or EBC) was derived from liquid sample measurements and compared to grinded sample (grist) measurements.

In general, the MIR data obtained in region 3027- 2787 (variable b) and 962-1218 cm^{-1} (variable f) showed the strongest correlation. The high correlation of variable (b) could be due to the signal of the stretching vibration (vC-H) of aromates like HMF or 1-Deoxyosone and 3-Deoxyosone (Ledl and Severin, 1978). Peak (c) could be designated to the asymmetric stretching vibration of carbon dioxide (vCO_2 at 2349cm^{-1}) (Groß, 2003) or possibly a little influence of aryl compounds (2309-2136cm^{-1}) (Gunzler and Gremlich, 2003), but due to the volatility of CO_2, no strong correlation could be found. The bands in range d (1807- 1567 cm^{-1}) might be due to many different stretching vibrations of carbonyl compounds like ketones, acids, esters, amides, etc. (Gunzler and Gremlich, 2003). The deformation vibration of water (δH_2O) was also located in that region (Gunzler and Gremlich, 2003) and might vary due to the different roasting degrees of the samples. Based on the simultaneous complex reactions during Maillard reaction, the composition and degradation of compounds could result in a non-distinct overlapping of vibrations. In the fingerprint region below 1500 cm^{-1}, each compound itself had a unique combination of absorption bands due to deformation vibrations generated by C-C and C-O interactions in ethers, alcohols, esters, etc. or stretching vibrations from C-N interactions from amines, amino acids or amides (Schmidt, 2000). Variable (f) (1218- 962 cm^{-1}) was located in that range and designated to stretching vibrations (vC-O) of polysaccharides resulting from ethers or carboxylic acids, like aldoses, or melanoidins (Cämmerer and Kroh, 1995) or the stretching vibration of ketones (v_{as}C-C) (Gunzler and Gremlich, 2003). This could explain the high correlations of variable (f) to reference factors, like colour values. During the roasting process of malt, sugars were degraded in Maillard reaction and other intermediates or melanoidins were formed (Cämmerer and Kroh, 1995).

Variable (f) could be designated to the stretching vibration of polysaccharides (vC-O), carboxylic acids

(aldoses), alcohol (vC-O), esters, and ethers as well as the stretching vibration of amines. Especially several bands in between 900 and 1200 cm^{-1} were assigned to the C1-0-C4 stretching mode of maltose. Sekkal et al. observed the 1-4 linkage in α-D-glucopyranosyl-(1→4)-D-glucopyranose at 922 cm^{-1} (Sekkal et al., 1995).

Conclusion

The study presented that developed partial least square regression models can be used as basis for implementation of process control via mid-infrared spectroscopy in future. HMF, color related and ESR values of the roasted malt in particularly are correlated with spectral data and therefore can be used as first approach in further studies. Most of the reference values use methods which require time-consuming sample handling and preparation. As a consequence, the data handling is done manually and it creates difficulty to implement in an automated process control. If a cost saving photometric approach measures only two selected ranges and utilizes both recorded time (h) and temperature (g) to enhance the prediction of a wanted reference value, an automated roasting of malt can be included cost effectively in future. By further enhancing the model, one or more actuating variables can be used for process control and as a basis to calculate a stop criterion of malt roasting. L*a*b*- values can be measured photometric and be standardized for each variety of the roasting process. With acceptable tolerance levels, e.g. confidence intervals of ∆E values, roasting of malt can be calibrated and taken advantage of for automatization.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest.

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Investigating cleaning in place (CIP) chemical, water, and energy use: A comparative study of standard operating procedure (SOP) for UK North East microbreweries

Harry Laing*, Steve Bull, Sharon Joyce and Chris O'Malley

School of Engineering, Merz Court, Newcastle University, Newcastle upon Tyne, NE1 7RU, UK.

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The effectiveness of multiple cleaning in place (CIP) procedures was observed from different local breweries in the North East of England. Experiments were also carried out to investigate possible reductions in chemical, water and energy use with regards to CIP, without compromising the effectiveness of the CIP performed. The effectiveness of CIP cycles was quantified using Hygiena's UltraSnap adenosine triphosphate (ATP) swabs, with a relative light unit (RLU) tolerance of <10-30 indicating a clean vessel. It is recommended that microbreweries use at least a 2% v/v dilution for caustic CIP cleaning cycles (based on a ~32% wt caustic liquor) for 35 min to ensure a thorough clean. High temperatures (40-60°C) did not indicate an improvement in cleanliness levels over ambient temperature water (10-20°C) over the 35 min cycle time. A single pass of 100 L of rinse water is adequate for vessels up to 1200 L to ensure removal of caustic residue and should be followed by a sterilisation stage. These recommendations are based on a final acid sterilisation cycle with 1% v/v dilution of a 5% wt Peracetic Acid (PAA) for at least 10 min. Reductions in the usage of caustic liquor, water and energy (heating) for caustic CIP cycles could yield microbreweries savings of over £1000 annually.

Key words: Cleaning, optimization, microbrewery.

INTRODUCTION

Brewing and beer production have been human activities since the beginning of urbanisation and civilization in the Neolithic period (Meussdoerffer, 2009). Over many years the brewing industry in the United Kingdom (UK) developed until it was dominated by a few large players with a relatively small product range. However, the UK no longer plays host to only a few 'powerhouse' brewing companies; as of 2018, there are now almost 2000 breweries in the UK (statista.com, 2020), and according to the Beer and Pub Association these are opening up at

*Corresponding author. E-mail: h.j.laing@ncl.ac.uk.

ORCiD: https://orcid.org/0000-0002-8783-2904

the rate of one every other day (Jones, 2015). Many of these are classed as microbreweries; a UK microbrewery is typically considered as a brewery that produces less than 15000 barrels of beer annually (Barron, 1995) *[*1UK barrel (bbl) is approximately 163L*]*.

It is imperative that all product is made in compliance with the regulations of the Food Standards Agency (FSA), in the UK, ensuring that beer produced is not contaminated and is fit for human consumption. Cleaning Techniques in the brewing industry vary widely, and there is no set procedure that is considered optimal, especially within the microbrewing community. Breweries tend to develop their own in house standard operating procedures with a lot of this handed down by word of mouth. Local microbreweries in the North East of England tend to perform cleaning based on an 'it has always worked' principle, without necessarily considering the potential reduction in water or chemical usage, unlike some of the larger breweries. Cleaning as a topic brewers are expected to know about, but too often 'will not admit to having a small amount of potentially prejudiced knowledge' (Boulton and Quain, 2006).

In addition to FSA regulations, brewers would be disappointed to find their product contaminated with unwanted microorganisms and compromised in flavour due to lack of brewery hygiene (Davies et al., 2015). Whilst the alcohol produced causes an inhospitable environment for most microorganisms, it is widely known that there are select few that can survive in the environment (Davies et al., 2015). The presence of contaminating microorganisms can affect product yield and beer flavour which, if passed to customers, can impact profitability (Davies et al., 2015; Hill, 2009). For microbreweries, local reputation could also be damaged, a potentially catastrophic outcome. Once the boiling of the wort and the fermentation stages of the brewing process have ended and the vessels drained, there remains on the interior of the vessels and in the heat exchanger microbiological and organic residue. This tends to be a combination of yeast, hops, some bacteria and any other ingredients/adjuncts used in the brewing process (such as fruit). There may also be inorganic residues (scales) from the hard water chemicals and beer stone (calcium oxalate and proteins) which is largely due to reactions between the alkaline cleaners used, hard water minerals and proteins in the beer. To prevent cross contamination from batch to batch, the vessel should thus be cleaned thoroughly.

The Standard Operating Procedure (SOP) for most breweries is to use Cleaning in Place (CIP) to clean vessels, a process thought to have been established by the dairy industry to provide adequate cleaning without the need to dismantle equipment (Meyers, 1959). CIP involves the pumping of various liquids through a spray ball to remove debris and microorganisms, and sterilise the equipment for future use. It is advisable that high shear is provided from the spray ball to encourage the displacement of biofilms and debris; a 1.5-3.5 m^3/h flow rate per metre of tank circumference is recommended (Boulton and Quain, 2006). It is generally accepted that there are 4 main phases to the cleaning of brewing equipment to ensure a thorough clean: pre-wash rinse, detergent (typically Caustic based) wash, rinse and sterilisation (typically acid) wash (Figure 1). An additional acid cycle and rinse is occasionally performed after the alkali rinse as a de-scaling measure. On some larger scale breweries the acid sterilisation stage is replaced with a sterilisation in place (SIP) procedure, using (sterile) steam to create a sterile environment (Davies et al., 2015). The pre-wash is used to remove loosely bound soil, alkali chemicals to remove organic soils and acids are used to remove inorganic soils, mineral scales (Goode, 2012) and sterilise the vessel, and the final rinse is to remove any alkali or acid from the vessel.

Typical guidance on the concentration of an alkali wash is to use Sodium Hydroxide (NaOH) at 2-5% w/v (Boulton and Quain, 2006), however, there have been drives towards a change to procedures in recent years due to price changes in chemicals. Traditionally, a hot caustic solution of 2-4% w/v was used for cleaning, but with increasing prices, the approach was changed to save costs and 1-2% w/v concentrations of caustic are now advised for the alkali wash for stainless steel vessels (Miller et al., 1960). It is generally accepted that no one material has all the desired qualities of a good detergent, but the detergent of choice is usually a mixture of different chemicals, with the primary chemical being Caustic Soda (NaOH) and possible sequestrant additions 'to improve emulsification and rinsability' (Miller et al., 1960).

Heineken NV, a large scale brewery, performs an ambient rinse, a hot caustic wash at 65-70°C, and then an intermediate water disinfection, using a 2% w/v concentration of caustic (Goode, 2012). However, Heineken suggest that lower CIP temperatures and chemical concentrations could be used in the UK to achieve the same level of cleanliness. The potential optimisation of detergent (caustic) use at Heineken was investigated, with results showing that concentrations of NaOH >1% w/v do not improve cleaning results, and therefore it is not cost effective to use higher concentrations than this. It was therefore advised that 1% w/v concentrations should be employed (Atwell et al., 2017), which is equivalent to 3% v/v dilution (Appendix A for conversion). Currently some microbreweries use less than 3% v/v NaOH, therefore there is scope to investigate further potential consequences.

In contrast, a pharmaceutical company will require a higher sterile level of cleanliness, but the CIP methods used may still be useful for the brewing sector. A pre-rinse of approximately 5-6 min is usually sufficient, and then a 1% (w/v) solution of sodium hydroxide at 75-80°C should be circulated for 15-20 min (Chisti and Moo-Young, 1994), supporting the premise that the reduced

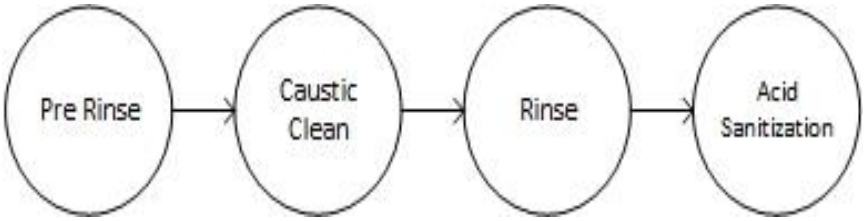


Figure 1. Generic CIP operating procedure for cleaning of brewery vessels.

concentration NaOH solutions can still provide adequate cleaning.

A similar study investigated the CIP regimes of yeast removal post fermentation for large scale breweries. The results found that a visibly clean surface could be achieved using 0.2 and 2% wt caustic based solution at temperatures ranging between 20-70°C (Goode et al., 2010); however, the largest removal of yeast film in the shortest time was recorded at 50°C, which suggested that the current operating procedure of the brewery (2% wt at 70°C) could need reductions of both temperature and chemical concentration (Goode et al., 2010).

A study on two American breweries, with the same SOP, identifies that they perform a soft rinse, a caustic cycle at 130°F (54°C) in half hour bursts, followed by a water rinse and then a sanitizing cycle of peracetic acid for 10 min (Deraney et al., 2015), where the tank is then considered clean. The suggested temperature for this procedure seems lower compared to that of other publications which typically suggest high temperatures of 60+°C. Above 60°C a significant steam and caustic vapour is produced which is unpleasant to work in (a problem with open top non-sealed vessels), which is another incentive to reduce the temperature of caustic cleaning solutions if possible.

However, it may be possible to compensate for the low cleaning temperature by increasing the cleaning agent concentration, and/or the flow speed (Praeckel, 2009), or on a microbrewing scale the temperature may be less important than previously considered. 'The Handbook of Brewing' advises the cleaning of fermentation and storage tanks from 'cold' to 40°C, but temperatures of 70-90°C for the cleaning of lauter tuns, mash tuns, wort coolers and all pipelines (Praeckel, 2009). This suggests that colder caustic CIP procedures could provide adequate cleaning potential.

It is apparent that there is no single identified best procedure for CIP in the brewing industry: the types of rinse and wash, chemicals used (and concentrations thereof), volumes of liquid, duration and temperature of the washes differ from brewery to brewery. To optimise the effectiveness of the CIP procedures, an optimum (or minimum) combination of temperature, volume of liquid and concentration of chemical needs to be sought. In addition, it will be necessary to define a method for measuring the cleanliness of vessels after the cleaning is done (and therefore compare the effectiveness of the CIP). This is the purpose of this study.

Previously mentioned studies tend to measure cleanliness of fermenters through visual inspections only. This study aims to use a more quantitative measure of cleanliness levels post cleaning to determine the effectiveness of different CIP regimes. To compare the different cleaning procedures in the breweries, a measurement technique for cleanliness was required. According to the FSA, all equipment that touches food must be 'cleaned effectively and… disinfected frequently', be kept in 'good order, repair and condition', and all chemical additives should be used in 'accordance with good practice' (FSA, 2013). Barron (1995) discusses the concept of Hazard Analysis and Critical Control Points (HACCP). The HACCP system was developed by the World Health Organisation and is the standard used throughout the EU Food Industry and is recognised by several legislative bodies (McCrimmon, 2004). The document discusses how beer is generally thought to be a safe beverage, but possible contamination should still be considered to ensure it does not occur. Whilst the document provides information on why beer can be considered safe (e.g., alcohol content and low pH inhibit bacterial growth) there is no information contained within it as to the procedures for CIP to ensure equipment is cleaned. The document advises that brewers should know the influence of temperature on microorganisms, but no further detail is presented. There is also no indication as to the level of cleanliness that should be achieved.

Currently, there is no defined legal quantitative 'standard' to which all breweries should comply, and this in turn results in different methods of cleaning practice from brewery to brewery. In addition, there are no guidelines for selection of a cleaning process and ensuring it is operated correctly. It also appears that very little action is taken in the way of regular testing for cleanliness by breweries (Moretti, 2013); this is likely due to the expense of the equipment and testing procedures such that small-scale brewing companies are reluctant to repeatedly test their equipment to see how clean it is. There are multiple ways in which cleanliness can be measured and quantified: for microbiological substances ATP Bioluminescence, the Direct Epifluorescence Filter Technique (DEFT) and Antibody DEFT are just some of

the ‘rapid’ methods for testing (Priest and Stewart, 2006), and for the chemical residue of the cleaning products themselves, conductivity and pH tests on the vessel walls and of effluent rinse water can be used (Chisti and Moo-Young, 1994).

The ATP technique measures living organic residual matter that may remain after a surface, device or piece of equipment is cleaned. A simple swab is taken of a surface area, and a handheld scanner provides a reading of Relative Light Units (RLUs). Whilst this method cannot specifically identify what live organic matter is on the tested surface, it provides a fast and easy method of estimating the overall condition of the vessel and plant and can be used as a rapid ‘go/no go hygiene check (Thomas, 2010). The ATP bioluminescence method is in use in many breweries for hygiene monitoring, product quality control and validating CIP performance in real time (Storgårds, 2000; Boulton and Quain, 2006).

Regarding ATP tolerance values for this study, the Hygiena’s ‘EnSURE’ bioluminescence reader was available, using their UltraSnap swabs that can be used on a wet or dry surface. For this study, Hygiena’s recommended tolerance of less than 10-30 RLU was implemented; a reading of <10 RLU indicates an ideally clean surface, and <30 RLU indicates a surface is clean enough for use but further improvements to cleaning could be made. It is also advised that, like the rest of the food and beverage manufacturing industry, brewers use industry accepted critical limits of 10 and 30 RLU (Hygiena, 2014).

CIP procedures used in the brewing industry vary from brewery to brewery, with no stipulated method. Varying temperature, duration and chemical concentration can yield different cleaning results (Goode, 2012). There is a gap in the knowledge of the microbrewing sector with regards to what is considered the ‘optimal’ CIP procedure. Further research will be undertaken into the effectiveness of the different cleaning techniques used in industry, posing the question of whether there is an optimal combination of temperature and concentration of detergent that offers optimal cleanliness.

In this study we compare the different cleaning procedures implemented by ten microbreweries situated on the North East of England and perform further experimental cleaning procedures on industrial sized brew kits in an attempt to determine if they can possibly reduce water, chemical and energy use. These breweries were chosen due to their various sizes within the microbrewing industry, their willingness to take part in the study and they were within reasonable travelling distance. The study focuses more on the cleaning of fermentation vessels, as this stage of the process presents the highest risk of contamination of the wort due to the operating temperature and sugar rich solution providing ideal conditions for bacterial growth. The exact method implemented by each brewery is presented in Table 1; the data has been anonymised and the ten breweries are known by the letters A-J and they range in size from 100 to 3200 L brew kit capacities.

METHODOLOGY

Taking ATP swabs

ATP swabs were used as the primary monitoring tool to determine how clean a vessel was before and after a clean. The Hygiena ‘EnSURE’ monitoring system was used to measure ATP samples, using the Hygiena UltraSnap™ surface swabs (which can be used to test a wet or dry surface). The following procedure, outlined in the EnSURE operational instructions, was used to obtain ATP sample swabs:

(i) For a flat surface, rub the swab over an approximate 4x4 inch area, rotating the swab repeatedly to ensure good coverage.
(ii) For irregular surfaces, such as outlet taps, valves or probes, ensure the area is swabbed thoroughly, rotating and using the same technique each time.
(iii) Return swab to holder, ensuring no contact with any surface.
(iv) Once swab has been taken, take reading within 4 h.

For each of the breweries visited, ATP swabs were taken before and after the full cleaning procedure were completed. Swabs taken prior to cleaning were only to ensure that a vessel was sufficiently ‘dirty’ before cleaning took place (typically providing RLU readings greater than 1000), but these tests are not reported here. After the cleaning SOP was completed, swabs were taken in various locations inside the vessel. The main focuses for swab locations were:

(i) The Krausen Line*;
(ii) Interior side wall of vessel (lower than the Krausen line); and
(iii) Any ‘Other’ difficult to clean areas, such as Welded joints, Valves or Probe Inlets.

**The interface between liquid and vessel where foaming occurs. Fermentation usually leaves a crusty residue in this area due to the typical ‘top fermenting yeasts’ used. Figure 2 gives detail of .the areas inside of the fermenter.*

A vessel was considered “clean” if the ATP swab reading was below 10 RLU and considered clean enough to use if less than 30 RLU, but improvements to cleaning techniques could be made. If the RLU was above 30, the vessel was not considered clean. Hence, the tolerance for determining whether a vessel is clean or not, for this study, was <30 RLU, as advised in the Hygiena ‘EnSURE’ operating instructions, however, <10 RLU is more desirable.

Cleaning procedures at external breweries

As part of this research, ten microbreweries in the North East of England were visited to observe their cleaning procedures in the hopes of optimising their cleaning regimes. The breweries that were visited as part of this study each had their own SOP for cleaning their vessels. ATP swabs were taken just after a pre-rinse, prior to cleaning, to ensure areas were sufficiently dirty and after the full CIP cycle to determine cleanliness. Each brewery used the generic SOP for cleaning shown in Figure 1 (with the exception of one that does not practise CIP but hand scrubs each vessel) with variations in Caustic concentration, temperature, duration and rinse water volumes. The actual procedures for each brewery are summarised in Table 1.

Table 1. Cleaning regimes of North East microbreweries in this study.

Brewery	Size of tank observed (L)	Conc. caustic solution (%v/v)	Volume of caustic solution (L)	Caustic duration (mins)	Temperature (°C)	Rinse water (L)	PAA dilution ratio (%v/v)	PAA Duration (mins)
A	1600	2	100	60	ambient	300	1	10
B	900	2	50	45	Ambient	150	1	10
C [1]	1600	~15	~10 - 20	N/A (hand scrub)	"Hot" (uses boiling water)	"Few minutes with hose"	0.5	0 (Splash with jug)
D	3200	2 [2]	~200	20	Ambient	600-900	0.1	10
E	900	2	100	25	Ambient	100	1	10
F	3200	2	200	30-45	Ambient		1	10
G	650	2	100	30	Ambient	400	1	30
H	100	1.5-2.5[3]	30	60	75	80-90	1	15
I	3200	2.5	100	60 (x2) [4]	70, then ambient	80	1	N/A (2 x 80L single run through)
J	450	3	100	20	40-60	300	1	10

1. Also includes a Nitric Acid rinse (a splash over with a jug) before the PAA rinse.
2. Usually only 1% v/v (2L in 200L) is used, but on this occasion, it was 4L due to the extra adjuncts added for fermentation.
3. Caustic concentration depends on whether cleaning wort or fermented beer from vessel. Observed cleaning regime for both instances.
4. Left to soak overnight after initial circulation, and recirculated next morning again for an hour.

In this study, the concentrations of cleaning solutions are reported in % v/v; for example, a 2% v/v caustic solution refers to 2 L of raw caustic liquor per 100 L cleaning solution. Although the branding of caustic liquor varied between some of the breweries, the % w/v concentration recorded on the Material Safety Data Sheets (MSDS) of each liquor used was the same (~32% w/v). Some liquors contained anti-foaming agents, which is not believed to impact the overall cleaning performance of the caustic liquor. Peracetic acid (PAA) is exclusively used for the Acid Sterilization stage, and each brewery purchases PAA at the same raw concentration (5% w/v), regardless of branding. Typical manufacturer advice is to use 5-10 ml PAA per litre of water (0.5-1% v/v dilution) to form the acid sanitizing solution.

Optimising the caustic cycle experiments

To optimise the cleaning procedures, further CIP tests were carried out on two industrial microbrewery kits: one with 450 L fermenters and the other with 900 L fermenters. These tests focused on the caustic cleaning cycle of the CIP regime outlined in Figure 1, to determine the effects of reducing temperature, duration and concentration of solution on the cleaning power of this stage.

The following steps were taken for the experiments performed:

(i) For each experimental run, the volume of rinse water and acid sterilization stage remained constant (a 100 L rinse following the caustic cycle, followed by a 10 min PAA cycle at ambient temperature and 1% v/v dilution).
(ii) The temperature was varied from ambient temperature water (typically 18-21°C depending the time of year) up to 60°C, the recommended maximum temperature for caustic solutions.
(iii) The chemical concentration of the brewery regimes observed were either 2.0% v/v or higher. Hence to determine a lower optimum, concentrations tested ranged from 0.5% v/v to 2.0 % v/v, increasing in steps of 0.5% v/v. The 0.5% increments were used as, typically, the breweries studied used half litre increments for measurements.
(iv) ATP swabs were taken throughout the duration of the caustic cycle (flow was paused momentarily), and the cycle was stopped if the RLU was within tolerance during this phase or until 60 min had passed, whichever came first. According to the breweries visited, as time is a precious commodity for brewers, leaving a tank on CIP for over an hour would be undesirable. Final RLU values stated in this study were taken after the PAA cycle.

RESULTS AND DISCUSSION

Comparative study

Table 2 shows the results of the average RLU swab tests taken at each site, and for some breweries this will be across several fermenters. Although cleaning regimes vary widely in duration, temperature, water and chemical usage, most breweries were able to meet the 10-30 RLU tolerance.

In general, swabs taken around the Krausen Line and Other areas (e.g. welds, scratches, thermowells, sample points) tended to have a higher RLU post clean, as shown in Table 2;

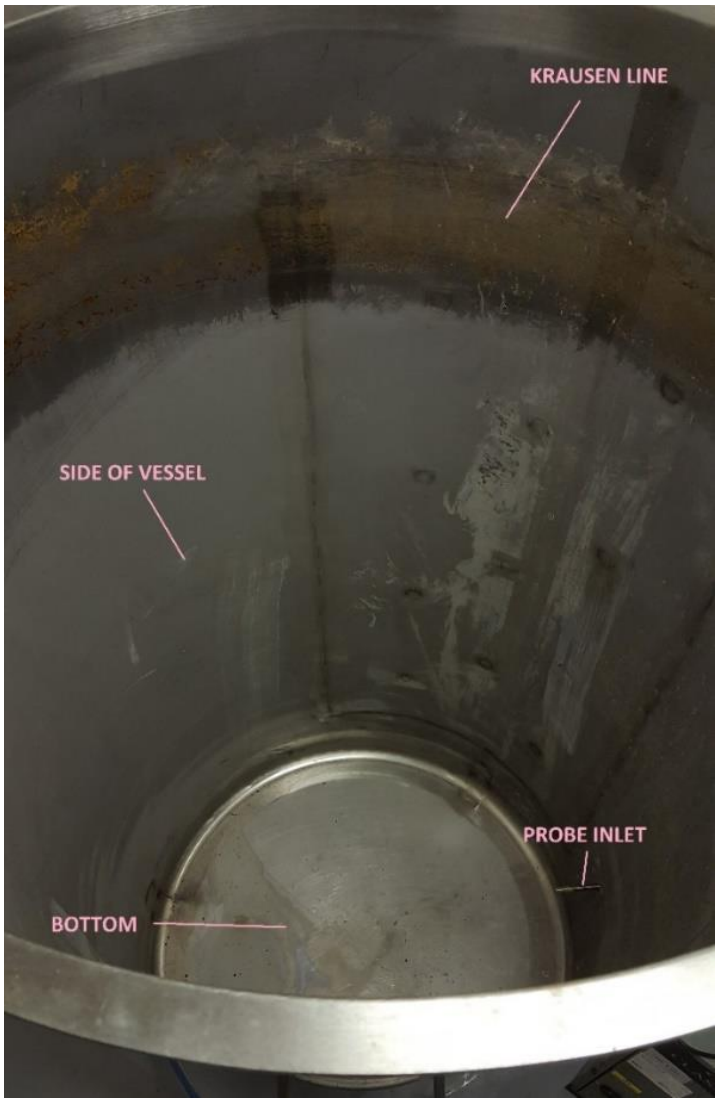


Figure 2. Inside of a typical cylindrical microbrewery fermenter after fermentation, with a specific highlight on the Krausen, Interior side wall and an example 'Other' area (Probe Inlet).

these areas are harder to clean, with larger deposits of biomass in the Krausen and the rough uneven surfaces allowing for increased accumulation of organisms compared to the smooth surface of the interior wall. Particular care and attention to these areas should be taken when cleaning fermentation vessels, to prevent possible accumulation of unwanted organisms that could lead to product contamination.

A notable observation from all breweries observed is the regime employed by Brewery C, who do not have a CIP cycle on their vessels. On two separate cleans of two separate vessels, the RLU swabs did not meet the cleanliness tolerance.

Thus, it is arguably better and more consistent to perform CIP in fermentation vessels than it is to manually scrub them. However, it should be noted that Brewery C has not yet encountered any contamination issues in their products. It was noticed that on some occasions, flow of caustic solution through the spray ball was not very high, and this resulted in poor cleanliness levels (that is, Brewery B's results). Whilst high flow rates can cause leakage or drips from the top of 'open top fermenters,' it is recommended that breweries ensure the spray ball pressure for their CIP regimes is sufficiently high to reach all areas of the fermenter with reasonable shear force. It is believed that the higher the shear force through the spray ball, the better removal of contaminants a CIP regime can provide.

Some of the breweries heat their CIP solution to high temperatures as it is believed to provide better cleaning results by the brewery staff. In contrast, Brewery A and Brewery E's results show that low temperature and lower

Table 2. Average RLU Results for each brewery.

Brewery	RLU Reading (±5%)		
	Krausen	Interior Wall	Other
A	1	-	5
B[1]	16	22	25
C[2]	148	35	28
D	-	7	63
E	2	1	1
F	67	6	-
G	0	1	-
H	-	0	0
I	2	106	1
J	2	0	7

Due to the shape and size of some vessels, full swabs were not possible for all vessels. 'Other' refers to typical problem areas, such as Valves, Probes or Welds. [1]Poor shear strength on side of vessel due to poor flow rate; [2]non-CIP clean.

caustic concentrations can achieve just as high levels of cleanliness, in comparison to those of Brewery H and Brewery J's regime and results. Although the brewery cleaning procedures of the study vary, the comparable high levels of cleanliness lead to the hypothesis that some breweries could reduce their chemical, water and/or energy usage, and have little to no impact on their cleanliness procedures. This would lead to long term potential savings for the brewery, but also a reduced environmental impact through use of less resource and reduced chemical loading on wastewater discharge. It was on this basis that further cleaning regime tests were carried out to investigate the impact of lowering chemical dosage, duration and temperature of the caustic cycle.

Further caustic cycle experiments

The main focus of the optimisation experiments was on fermenters that had a high amount of yeast, however, a few low concentration tests were performed on Bright Tanks (tanks that typically hold 'clean beer' free from yeast and hops, and on occasion can also be Carbonation tanks). The experiments performed are summarised in Table 3, and show whether the experimental setup achieved the cleanliness tolerance or not. Swabs were still taken from multiple locations (Krausen, Interior Wall and 'Other' areas) inside the fermenter, and cleanliness was achieved if all three areas swabbed were within tolerance. The measured RLU values from all the fermenter-based experiments are shown graphically on Figure 3, with the RLU tolerances of 10 and 30 shown as the Green and Red planes, respectively.

The results from Table 3 indicate that low concentrations of caustic (1.0% v/v) would be adequate for the cleaning of Bright beer tanks at low temperatures in as little as 10 min, provided that the tank has not had any micro-bacterial additives (such as bottling yeasts) and is purged of CO_2 prior to the commencement of cleaning. However, further investigations into the cleaning of Bright tanks should be performed before a formal recommendation to use such a low concentration. As a note of warning for using NaOH to clean carbonation vessels, CO_2 and Caustic react together with the resulting reaction causing a vacuum which could lead to vessel collapse (Boulton and Quain, 2006; Manzano et al., 2011) if left fully sealed.

Table 3 and Figure 3 show clearly that, for fermentation vessels of at least 450 L in size, a caustic solution of concentration 1% v/v is inadequate to provide consistent acceptable levels of cleanliness, and a solution of 0.5% v/v cannot reach the tolerance values, regardless of the temperatures or duration of the cycle. It would suffice to argue that the concentrations of 1% v/v and lower of caustic is too weak to ensure the removal of all microbial growth within the vessel. A concentration of 1.5% v/v, however, consistently reaches the tolerance requirements after around 30-40 min of CIP, with 2% v/v reaching tolerance within 20-30 min. At lower concentrations of caustic solutions, increasing the temperature of the solution did not provide an increase in cleaning power, contrary to the expectations of the local brewers.

Optimal caustic cleaning cycle

When examining all the cleaning regimes used as part of this study (industry observed or experimental, Figure 4), it is evident that the most important variable when deciding on a caustic cleaning SOP is the concentration of the caustic solution. Caustic solutions of 0.5% v/v were unable to meet the required tolerances and should not be

Table 3. List of further experimental procedures and results to test lower temperatures and lower concentrations of caustic cleaning solutions. Caustic cycle was stopped once RLU tolerance was achieved or once 60 min had passed.

Concentration (% v/v Caustic)	Duration of caustic cycle	Temperature (°C)	Vessel type	Tolerance achieved?
2	30	21	Fermenter	Yes
2	25	21	Fermenter	Yes
2	25	25	Fermenter	Yes
2	10	21	Fermenter	Yes
1.5	30	19	Fermenter	Yes
1.5	35	18	Fermenter	Yes
1.5[1]	55	40	Fermenter	Yes[1]
1	60	40	Fermenter	No
1	60	60	Fermenter	No
0.5	60	19	Fermenter	No
0.5	40	40	Fermenter	No
0.5	60	60	Fermenter	No
2[2]	60	19	Carbonation/Bright Tank	No[2]
1[3]	60	18	Carbonation/Bright Tank	No[3]
1	10	19	Carbonation/Bright Tank	Yes
1	10	18	Carbonation/Bright Tank	Yes

[1]Test started out at 1% v/v, but by 40 min RLU had not reduced for 30 min (from 10-40 min), thus concentration was increased to 1.5% v/v at 40 min (adding 500 ml caustic liquor). [2]The tank had not been purged of CO_2. The CO_2 reacted with caustic; hence the cleaning power was reduced drastically. [3]The Carbonation Tank had 'Bottling Yeast' inside to allow for bottle conditioning of beers, so was not clean beer.

employed for cleaning of fermentation vessels. Similarly, whilst 1.0% v/v concentrations can achieve the cleanliness tolerance, solutions of this strength are unable to do so consistently, even at warmer temperatures.

Once a concentration of at least 1.5% v/v has been reached, the results indicate that the overall cleanliness of vessels does not improve with increased temperature; non-heated (ambient) temperature water is perfectly acceptable for cleaning at this concentration. Increasing the concentration beyond 1.5% v/v still provides cleaning results within tolerance but does show more RLU values below the 10 RLU threshold.

Increasing the concentration from 2% to 3% v/v does not appear to provide any significant benefit regarding the cleanliness of vessels, even with the increased temperatures observed in industry. The duration required to ensure that cleaning reaches tolerance does reduce slightly when the concentration is increased from 1.5-3% v/v regardless of temperature as follows: 30-40 min for 1.5% v/v, 25-35 min for 2.0% v/v and 20-30 min for 3.0% v/v.

Based on the results shown in Figure 4, it is sufficient for a brewery to employ a caustic CIP cycle of 1.5% v/v concentration for at least 40 min at ambient temperatures to ensure a thorough clean, providing there is adequate sheer force on the inside of the vessel and the raw liquor is at least 32% w/v. However, to provide a better clean (under the lower RLU tolerance) a caustic solution of 2% w/w for at least 30 min is recommended for fermentation vessels. Although there are a few instances where higher concentrations do not meet tolerance, these could be explained through non-optimal cleaning conditions, such as reduced spray ball shear force or a lack of attention to the manual cleaning required for difficult places to clean, such as sample taps and valves.

Potential caustic savings

Most breweries observed used a 2% v/v dilution of caustic for their cleaning procedures, but some used higher amounts. For a microbrewery, the difference between using 2 and 3% v/v in caustic can result in reasonable savings in chemical usage (e.g., for 100 L, it is 1 L of caustic saved per cleaning cycle). Lower chemical doses also help with compliance of chemical discharge into sewers. At the time of writing, a 25 L Caustic Liquor drum could be purchased for £26.45 (Murphy and Son LTD, 2018). Assuming a brewery does CIP six times a week (312 cleans annually) and uses 100 L of water per cycle, a 2% v/v dilution would require 26 drums of caustic a year, whereas a 3% v/v would require 39 drums, a difference of £344. However, other caustic brands can cost as much as £37.50 a drum (Niche Solutions, 2020), which would increase savings to £488 annually.

Similarly, providing there is no compromise on the cleanliness of the vessel, reducing the volume of caustic

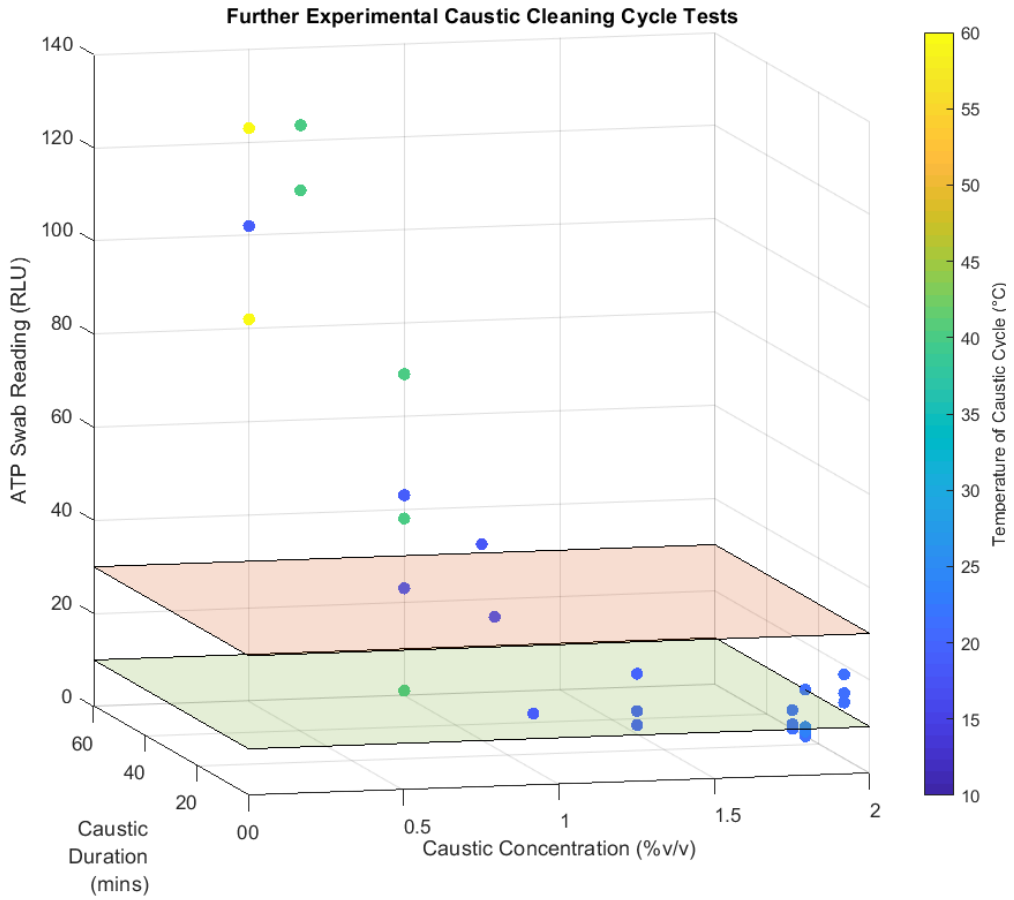


Figure 3. ATP swab RLU values for each additional experimental result for the fermenter-based experiments. Green and Red planes show the RLU tolerances of 10 and 30, respectively.

solution from 200 to 100 L per cycle would half the caustic costs annually; although some of the larger breweries observed used 100 L of caustic solution, it is important that breweries ensure the volume of caustic solution used does not cause pumps to run dry on occasion if reducing the volume.

Potential rinse water savings

It was witnessed that large volumes of rinse water are used in some breweries compared to others, such as the comparison between Brewery D and I, where brewery D uses over 500 L more to rinse vessels of a similar size. Regardless of the volume of caustic used for a rinse, the amount of caustic left in a vessel after draining it is reflective on the size of the vessel; the film left on the vessel walls should be all that remains. Thus, there seems to be little justification for extra water usage.

Figure 5 shows the results of a pH test on initial and final samples of rinse water used when rinsing a fermenter, carbonation tank or brew Kettle with a connected heat exchanger. These results indicate that, for at least up to 1200 L vessel, 100 L of rinse water should be adequate to ensure no caustic residue is left inside the vessel, with an additional 100 L required for a heat exchanger.

According to Northumbrian Water, the local water company to the area, the cost of a cubic meter of water (1000 L) is 106.46p (Northumbrian Water, 2020). Again, assuming a brewery performs 312 cleans annually, a 500 L saving of rinse water per clean would result in a cost saving of £166. Breweries must also pay per cubic meter of sewerage, at 77.39p (Northumbrian Water, 2020), resulting in an additional saving of £121 in sewerage charges. Hence a 500L saving in rinse water per clean, based on six cleans a week, could result in a saving of almost £300 per year (£287 per year), as well as a reduced load on local sewerage systems and the environment.

Potential energy savings

Based on the findings of this study, adequate cleaning is provided by ambient temperature caustic solutions just as

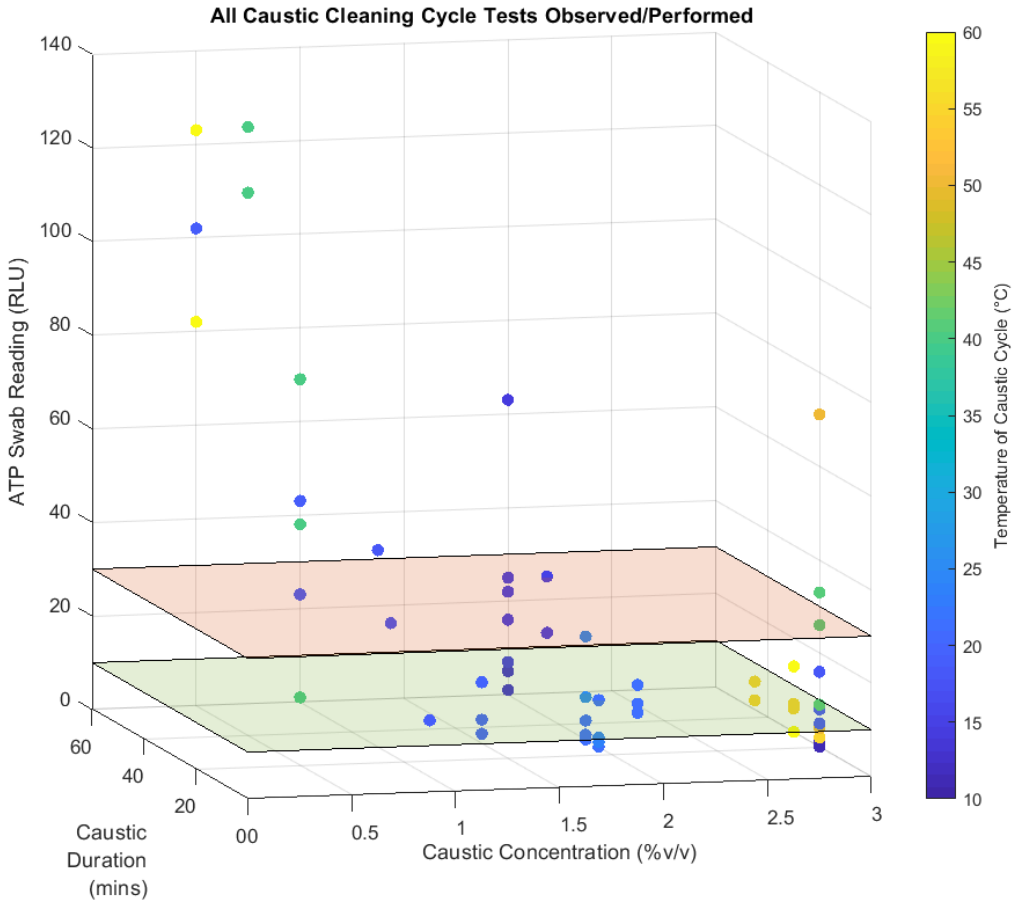


Figure 4. ATP swab RLU values for additional experimental fermenter-based results and the observed results from industry for breweries performing CIP with a PAA cycle of at least 10 min. Green and Red planes show the RLU tolerances of 10 and 30, respectively.

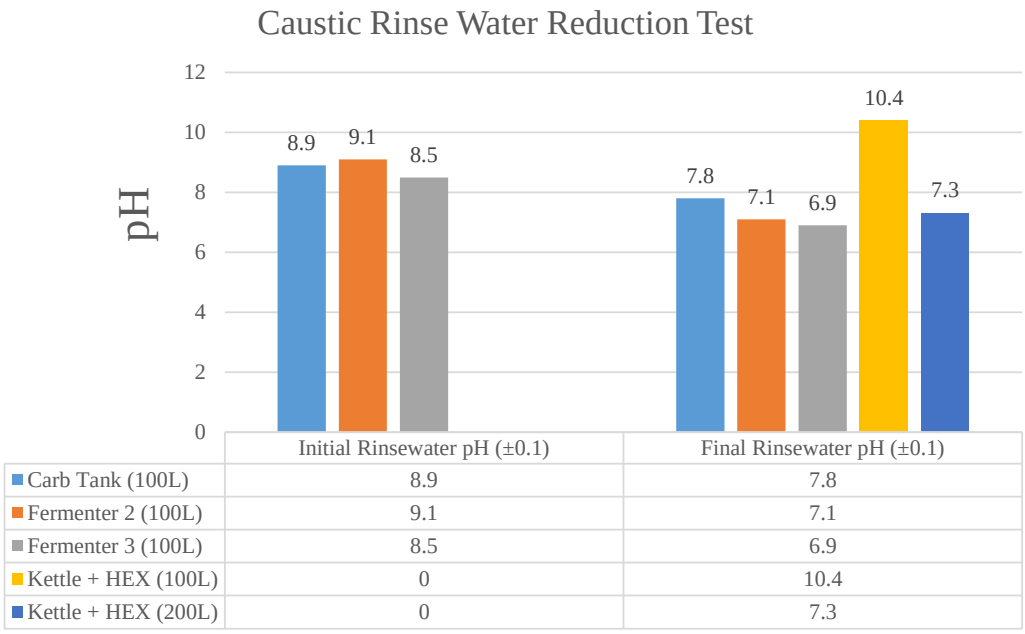
well as higher temperature solutions. This suggests that there could be no need to heat the cleaning solution, which could present significant energy savings for small business breweries.

As a rough estimate, assuming a 100 L cleaning solution requires heating prior to the addition of caustic, it will require approximately 18.9MJ of energy to heat 100 L from 15-60°C, which is equivalent to 5.25 kWh. Most breweries heat their water using electric immersion heaters. Based on the UK Governments published small business energy rates, electricity cost small businesses 14 p/kWh per quarter in 2019 (UK Government, 2020). As before, assuming 312 annual cleans of a brew kit, eliminating the heating of caustic cleaning solutions could save small breweries up to £230 in electricity costs annually. For breweries using 200 L of heated solution, the savings would double to £460.

Conclusion

Microbreweries must maintain high levels of cleanliness to ensure products are not contaminated, thereby avoiding reputational damage to their brands. It is reassuring that, in general, the microbrewery cleaning practices currently employed across North East England are considered acceptable and vessel cleanliness adheres to the RLU tolerance applied as part of this study. There is no single CIP technique employed by microbreweries in the North East that could be deemed ‘the best.’ Although some will feel peace of mind with SOP above the minimum requirements, based on the observations of this study, some breweries could be over using chemical, water and/or energy resources during cleaning. Thus, there is the clear potential for financial savings and mitigation of environmental impacts.

It is recommended that microbreweries use at a bare minimum 1.5% v/v dilution of caustic solutions during CIP of fermentation vessels for 40 min, though at least 2.0% v/v for 35 min is preferable to ensure a high level of cleanliness. Should a brewery prefer to use 3.0% v/v, then a cleaning duration of 30 min should suffice. This is assuming there is adequate shear force provided by the spray ball on the vessel surfaces. Reducing chemical



	Initial Rinsewater pH (±0.1)	Final Rinsewater pH (±0.1)
Carb Tank (100L)	8.9	7.8
Fermenter 2 (100L)	9.1	7.1
Fermenter 3 (100L)	8.5	6.9
Kettle + HEX (100L)	0	10.4
Kettle + HEX (200L)	0	7.3

Figure 5. Rinse Water test to see whether 100 L is enough water to fully rinse a 450, 600 or 900 L tank of caustic residue. Carb Tank is a 600 L vessel, Fermenter 2 is 450 L and Fermenter 3 is 900 L. The kettle is a 1200 L tank with a heat exchanger (HEX) connected in series. Volumes displayed next to names of vessels indicate rinse water volume.

dosage amounts for some breweries could provide annual savings of up to £300.

Heating the caustic solution does not appear to provide an additional benefit; breweries could use cooler or ambient temperature water without hindrance to cleaning results and achieve savings of over £200 annually in energy costs. At least 100 L of rinse water should be used for vessels up to 1200 L in size, including heat exchangers. Larger vessels were not tested as part of this study, but it is likely that 150 L would suffice for vessels up to 1800 and 200 L for vessels up to 3200 L. Although the cleanliness of vessels is of highest importance, work done as part of this study shows that microbreweries should still consider the potential cost savings of optimising cleaning regimes and that cleanliness levels do not necessarily have to suffer as a result of such optimisation practices. It should also be noted that this is only one measure of cleanliness; non-living organic residues are also important to consider, particularly for craft beers with non-standard additions.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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