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# Impact of colour adjustment on flavour stability of pale lager beers with a range of distinct colouring agents

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ABSTRACT

The impact of colour adjustment on the flavour stability of five pale lager beers with a range of colouring agents such as specialty malts, colouring beer and artificial caramel colourant was investigated. The research focused on determination of the endogenous anti-oxidative potential (EAP) of the beer samples using a novel Electron Spin Resonance (ESR) method. The results were correlated with the concentration levels of a portfolio of compounds formed during beer ageing, which were detected and quantified by GC–MS. The beer samples were also assessed by the ICBD sensory panel. Additionally, the quantification of organic radicals of the specialty malts and the roasted barley were conducted by ESR (whole intact kernel and milling fraction measurement). Based on the results of this holistic approach, a colouring agent was identified that enhanced the flavour stability of pale lagers based on the final beer's physical-, chemical-, and sensory-properties.



## 1. Introduction

Little research has been carried out on the influence of colouring agents on flavour stability of pale lager beers. However, contrasting findings have been reported by research focused on related issues. For instance, it has been found that the addition of melanoidins and caramel to lager beer, which was subsequently exposed to light, depressed the evolution of $H_2S$ (one indicator of light damage). Additionally, when the colour of lager beers was increased by using a colouring agent, the concentration of 3-methyl-2-butene-1-thiol (MBT), formed on exposure to light, was reduced (Sakuma, Rikimura, Kobayashi, & Kowaka, 1991). Nevertheless, recent studies have indicated that melanoidins and caramelisation products of caramels promote the oxidative instability of lager beers. The use of caramel colourant increased the levels of radicals in the Fenton reaction assay, indicating that caramel colour is able to accelerate metal-catalysed oxidation of beer. Traces of iron and copper commonly found in beer can act as catalysts in the radical generation during beer ageing (Nøddekær & Andersen, 2007).

Previous research has also demonstrated that fresh dark beers produced using varying ratios of dark malts resulted in elevated concentrations of flavour-active beer ageing carbonyls such as 3-methylbutanal, 2-methylbutanal, 2-phenylethanal and iso-butanal (Forster, Narziss, & Back, 1998). Moreover, the anti-oxidative potential of specialty malts does not appear to increase their colour intensity conferred on the beer. In fact, a higher anti-radical activity for the pale malts and crystal malts with intermediate browning degree per unit of colour in comparison to black malts has been noted (Cantrell & Briggs, 1996; Sovrano, Buiatti, & Anese, 2006). This assumes that the role of malts on improvement of beer flavour stability is dependent on the relationship between reducing power and colour.

In connection to this, it has also been reported that malts roasted at temperatures above 150 °C contain a lower anti-radical activity than malts of the same colour, which were roasted at lower temperatures for a longer period of time (Coghe, Gheeraert, Michiels, & Delvaux, 2006). This indicates anti-radical groups are involved in the advanced stages of non-enzymatic browning reactions, which are significant at temperatures above 150 °C. The maximum anti-radical activity seems to be related to the end-roasting temperature rather than to a specific malt colour. This is supported by other investigations that found that dark beers produced with a high ratio of dark colour malt (Munich style) showed better head retention and flavour stability in comparison to dark beers brewed with roasted malts. Additionally, dark colour malts kilned with a longer final kilning temperature (7 h) enhanced the flavour stability of dark lager beers in comparison to those kilned with conventional procedures (Preuß, Forster, Thum, & Back, 2001). Thus, the anti-oxidative activity of malt appears to be dependent on the time–temperature roasting programme.

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In this study, we sought to understand whether the mode of colour delivery in pale (lager style) beers had any significant impact on the final flavour stability of these beers.

## 2. Materials and methods

### 2.1. Beer samples

Five pale lager beers were brewed at the 2 hL pilot brewery at the International Centre for Brewing and Distilling (ICBD). Four of this portfolio of beers were colour-adjusted using different colouring agents (i.e. specialty malts and caramel colourant). An additional non-colour adjusted beer was brewed as control. Each grain bill for the colour-adjusted beers was designed to obtain a final beer colour of 7.5 (±0.5) EBC reference. Two-row "Optic" spring barley pilsner malt from Scotland (Bairds Malts Limited, Pencaitland, Scotland) was used as base malt for all the beers produced. All of the two-row "Marthe" spring barley specialty malts were provided by Weyermann Malzfabrik GmbH (Bamberg, Germany). Tables 1 and 2 present some relevant specifications of these specialty malt products and the ammonia caramel colourant (Beer caramel colour III), with a typical colour of 29,800 EBC and colloidal positive charge, provided by D.D. Williamson & Co. Inc. (Louisville, KY) (Caramel Products, 2007).

The grain load was comminuted by hammer milling. Brew liquor with a total hardness of 14.5°dH (±0.5°dH) (German hardness), a concentration of calcium ($Ca^{2+}$) and magnesium ($Mg^{2+}$) of 9.0 mg/l (±1.0 mg/l) and 14.0 mg/l (±1.0 mg/l), respectively, a carbonate hardness of 8.0°dH (±0.5°dH) and a residual alkalinity of 3.5°dH (±0.5°dH) was used. Table 3 shows the mashing specifications used for the production of wort of the samples under investigation. Wort filtration was carried out by a 35 kg working capacity-mash filter (MEURA 2001). Lixiviation liquor temperature was 78 °C (±0.3 °C) and the recirculation of wort for the initial wort clarification was achieved in less than 7 min. Minimum extract of last running was considered as 1°P.

The boiling time was 60 min. The bitterness specification of the beers was 20 International Bitterness Units (IBU). Hop dosage was performed at the beginning of the boiling process with Hallertauer–Magnum pellets (12.7% w/w α-acids), and Saaz pellets (6% w/w α-acids) were 10 min prior to the end of boiling. In the case of the locally-brewed beer colour-adjusted with caramel colourant, the addition of this colouring agent was conducted at this stage of the process. The separation of hot breaks (hot trub) was carried out for 10 min by means of a whirlpool system. The whirlpool rest time was no more than 20 min. The wort original gravity obtained was 12°P (±0.3°P). Bottom fermenting yeast "Saflager S-23" (Saflager-S23. Fermentis, 2007) slurry was pitched with a concentration of 15–20 × $10^6$ yeast cells/ml with viability up to 95%. First to fifth yeast generations were used for this experimentation only. No multi-filling method was applied and no antifoam agents were used either during the boiling or during fermentation. The primary fermentation period took five to seven days at 12 °C (±0.3 °C). The period allowed for the reduction of vicinal diketones was 48 h with the cooling system off of the fermentation tank. The beer maturation was held at 2 °C (±0.3 °C) and 0.8–1.0 bar for 14 days. The beers were colloidally stabilised with 50 mg/l of Lucilite TR (PVP coated-silica gel filter aid) (McKeown & Thompson, 2003) and filtered by means of a sheet filter with the use of Carlson XE400 filter sheets (0.5 μ) (Carlson Filtration Ltd. XE 400: Product specifications, 2007). The filtered beers were transferred into the bright beer tanks (BBT's) at 2 °C (±0.3 °C). No foam stabilisers were used. Beers were bottled with a filling volume tolerance of 1% and carbonated at 2.5–3.0 vol. by means of a CW250-G carbonating and counter pressure bottle filling equipment (CW250-R&D. Moravek. International Limited, 2007). The bottled beers were pasteurised at no more than 35 PU and stored in the dark at 4 °C (±0.5 °C). Aged beers were considered as beers with an ageing of 12 months while the forced aged analogues as beers thermally treated at 60 °C for 7 days.

**Table 2**
Artificial caramel colourant (CARAMEL #301) specifications (Caramel products. D.D. Williamson & Co. Inc.).

| Specifications | Values |
|---|---|
| Type of caramel colourant | Type III (ammonia caramel) |
| Colour IoB (typical) | 31,500 |
| Colour EBC (typical) | 29,800 |
| Hue index (typical) (H.I.) | 5.5 |
| Percent solid (%) | 66 |
| Specific gravity (kg/l) | 1.320–1.330 |
| Baume at 60 °F (15.56 °C) (°Baume) | 35.2–36.0 |
| pH, as is | 4.2–4.8 |
| Viscosity at 68 °F (20 °C) (Max. cps) | 4000 |
| Colloidal charge | Positive |

**Table 3**
Mashing specifications.

| Specifications | Values |
|---|---|
| Brew liquor/Grist ratio | 2.5:1 |
| Mashing method | Step mashing infusion |
| Mash-in temperature (°C) | 55.0 (±0.3) |
| pH-correction for brewing liquor | No |
| pH of mashing | 5.5 (±0.1) |
| Mashing programme | 55 °C for 10 min, from 55 °C to 62 °C at 1 °C/min, 62 °C for 60 min, from 62 °C to 72 °C at 1 °C/min, 72 °C for 20 min, from 72 °C to 78 °C at 1 °C/min. Total time: 113 min Mashing temperature tolerance: 0.3 °C |
| Mash-out temperature (°C) | 78 (±0.3) |

### 2.2. Standard analyses of the locally-brewed pale lager beers

The standard analyses of the locally-brewed pale lager beers were carried out in triplicate according to the official methods of analysis of the Mitteleuropäische Brautechnische Analysenkommision (MEBAK) and the European Brewing Convention (EBC) (Brautechnische Analysenmethoden. Band II. Mitteleuropäische Brautechnische Analysenkommission, 2002; Analytica-EBC. European Brewing Convention, 1997). In addition, a series of reducing power tests of the caramel colourant (Caramel #301) at distinct concentration levels (0.2% w/w and 0.4% w/w, respectively) in commercial pale lager beer and in distilled water were

**Table 1**
Specialty malts specifications (Weyermann Malzfabrik GmbH).

| Malt type | Moisture content (%) | Moisture content (%) | Wort colour (EBC) | Specific weight (kg/m$^3$) |
|---|---|---|---|---|
| CARAHELL® (Light crystal malt) | 9.0 | 74.0 | 20–30 | 580–640 |
| Melanoidin malt | 4.5 | 78.0 | 60–80 | N/A |
| CARAFA® SPECIAL Type III (Dehusked roasted malt) | 3.8 | 65.0 | 1300–1500 | 500–550 |