F.L. Steil, T. Schramm, C. Neugrodda, M. Gastl and T. Becker

The influence of water composition on the gushing behaviour of malt in Modified Carlsberg Test

Gushing was described for the first time in 1909 [24]. Since then, the overfoaming of carbonated beverages has been over the time intensively investigated. Different substances that cause or suppress gushing have already been analysed. However, the influence of water composition i.e. ions and their concentration on gushing behaviour still requires studying. In this research, different self-prepared waters and a gushing positive malt were used for the gushing prediction test Modified Carlsberg Test (MCT). During the tests the self-prepared waters were used instead of the MCT water. The research hypothesis is that ions, which are naturally present in drinking water, and in respective brewing water, react with substances that stabilize gas bubbles and result in a gushing inhibiting effect. The lowest overfoam amount (OF) was achieved when magnesium was present in concentrations of 30 to 60 mg/L. The overfoam amount in MCT was significantly reduced when water enriched with magnesium ions and several anions (chloride, sulphate and hydrogen carbonate) was used, compared with the used reference, Bonaqa® water. The findings obtained in the present research provide another piece of the puzzle enlighten the problem of gushing.

Descriptors: gushing beer, gushing test, modified carlsberg test, MCT, water quality, ions, magnesium, potassium

1 Introduction

Gushing is the spontaneous overfoaming of carbonated beverages after opening the bottle without any external mechanical influence, e.g. shaking. The specific phenomenon is considered a quality defect. The resulting return or recall actions can lead to high financial losses as well as image damage. The causes of gushing, which is a multi-causal problem [17, 39], are still not fully understood in terms of their biophysical mechanisms. It is known that gushing can be caused, on the one hand by raw materials (primary gushing) and on the other hand by technological factors in the production of beverages (secondary gushing) [19].

Over the years, researchers have aimed on the development of a reliable gushing prediction test [1, 2, 5, 7, 9, 10, 13, 14, 16, 20–22, 26, 28, 33, 34, 36, 38]. The Modified Carlsberg Test (MCT) [23] is established as a reference test, which is recommended by the MEBAK as an instrument for the early detection of gushing and is frequently required in supply contracts.

Concerning primary gushing, the fungal metabolites hydrophobins are seen as promoter of gushing, since they settle on gas-liquid surface and stabilize the CO_2 bubbles [12, 40]. Secondary gushing has many agents that contribute to it, e. g. non-specific lipid proteins (ns-LTPs) [4, 35], iron ions (Fe^{3+}) [6, 8], hops compounds like Tetrahop [27, 32], storage temperature[11] and bottle surface roughness [15, 35].

Beer has normally a water content of more than 90 % [4]. However, the influence of water components on gushing has been poorly researched, so far. The connection of calcium in form of calcium oxalate crystals and gushing is well known, especially regarding secondary gushing. The crystals can act as condensation nuclei for CO_2 if it's formed in bottled beer [3, 25, 41]. However, at primary gushing, its influence is still almost lacking. In previous work [18], a significant influence of water quality on the gushing behaviour could be shown: Monovalent sodium ions (Na^{+}) as well as divalent calcium ions (Ca^{2+}) reduced gushing when present in high amounts (> 600 mg/L).

Previous research [37] showed decreased or no overfoaming in samples with added sulphate ions and increased overfoaming with added chloride ions. Salts of the aforementioned anions were considered on the current research. The influence of cations like potassium and magnesium on gushing is unknown till now, although these cations are normally present in drinking water too. During beer production, drinking water is treated to become suitable for use in the brewing process, since its components affect, e. g., pH value and consequently enzyme activity.

In our study we want to investigate the influence of potassium and magnesium cations respectively in combination with one anion (chloride or sulphate) and in presence of more anions (sulphate,

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Authors

Fernanda Larissa Steil, Tim Schramm, Christoph Neugrodda, Martina Gastl, Thomas Becker, Institute of Brewing and Beverage Technology, Technical University of Munich, Freising, Germany; corresponding author: christoph.neugrodda@tum.de

chloride and hydrogen carbonate) on gushing behaviour in MCT.

The results expand our knowledge of the effect of water ions on gushing. If the ions naturally present in drinking water could possibly reduce the intensity of gushing, the adjustment of brewing water composition could become an easy and inexpensive way to contribute reducing this problem.

2 Materials and methods

In this research a pilsner malt tested in MCT by MEBAK [23], classified as gushing positive (OF = 141 ± 26 g), was used. Different waters with defined ion composition were used. Besides a reference, Bonaqa®, the waters were prepared by adding salts to fully demineralised water.

2.1 Production of the different water qualities

The mass of each salt was calculated applying the same scheme to all water compositions. The calculation for magnesium chlorite (water Am, 25 mg/L magnesium) is given as example.

1. The required amount of substance (n) per liter was calculated from the aimed final concentration of an ion (m) and its molar mass (M) [2.1].

$$n\ [\frac{mol}{L}] = \frac{m[\frac{g}{L}]}{M[\frac{g}{mol}]} \quad [2.1]$$

For magnesium chloride:

$$MgCl_2 \rightarrow Mg^{2+} + 2Cl^- \quad [2.2]$$

$$1\,mol \rightarrow 1\,mol + 2\,mol \quad [2.3]$$

$$n_{Mg^{2+}} = \frac{m_{Mg^{2+}}}{M_{Mg^{2+}}} = \frac{0{,}025\,[\frac{g}{L}]}{24{,}305\ [\frac{g}{mole}]} = 1{,}02859 \times 10^{-3}\,mol\ Mg^{2+}/L \quad [2.4]$$

2. The required mass of the salt as a pure substance per liter resulted of the calculated amount of substance per liter and the molar mass of the salt. Since one mole magnesium chloride dissociate to one mole magnesium ion and two moles chloride [2.3], the result is:

$$n_{Mg^{2+}} = n_{MgCl_2} \quad [2.5]$$

$$m_{MgCl_2} = n_{Mg^{2+}} \times M_{MgCl_2} = 1{,}02859 \times 10^{-3} \times 95{,}211 = 0{,}09793\ g\ MgCl_2/L \quad [2.6]$$

3. In a similar way, the content of chloride ion can be calculated:

$$n_{MgCl_2} \rightarrow 2 \times n_{Cl^-} = 2 \times 1{,}02859 \times 10^{-3}\,\frac{mol}{l} \quad [2.7]$$

$$m_{Cl^-} = 2 \times 1{,}02859 \times 10^{-3}\,[\frac{mol}{L}]\ .M_{Cl^-}\,\frac{g}{mol} = 72{,}93 \times 10^{-3}\ g\ Cl^-/l \quad [2.8]$$

4. In case the salt was present as dihydrate, the mass fraction of bound water was also considered:

$$m(salt * 2H_2O) = m(salt) + n(salt) * 2 * M(H_2O) \quad [2.9]$$

It should be noted that the use of salts involves an addition of cation for each desired anion addition and vice versa. The waters were filled in 0,33-L-Bonaqa-Bottles. The CO_2 contents of the waters were measured with AntonPaar CBoxQC At-line and the values were 6,5 ± 0,1 g/L. The ion contents were determined using ICP-OES (DIN EN ISO 11885:2009-09, [30]) for cations and liquid chromatography were used for anions (DIN EN ISO 10304-1:2009-07, [31]). ANOVA (0,05 significance level) was used for the statistical analysis.

2.2 Gushing Test

The MCT by MEBAK [4] was carried out with the self-prepared water instead of the MCT water. In order to exclude a possible influence of the production of wort, when more than one wort was produced, they were homogenised in a beaker before mixing with the waters. The results are shown as mean value of the overfoam amount (OF) of three bottles in gram [g] and its standard deviation.

3 Results and discussion

The total ion concentration of the waters varied from 98 mg/L to 1299 mg/L. Bonaqa® water was considered the standard for MCT by MEBAK [4] until 2022. Then it was replaced by MCT water [10]. Table 1 shows the ion compositions in tested waters (measured ion concentrations of Bonaqa®, C1 and calculated ion concentrations for the others waters).

The influence of cation (water A1 to D1) and anion (water E1 to H1) on gushing was investigated. To generate a comparative value, Bonaqa® test water (Bo) was also used (OF = 141 ± 26 g in MCT). Figure 1 shows the results of the MCT performed with the gushing-positive malt and waters with different ion composition.

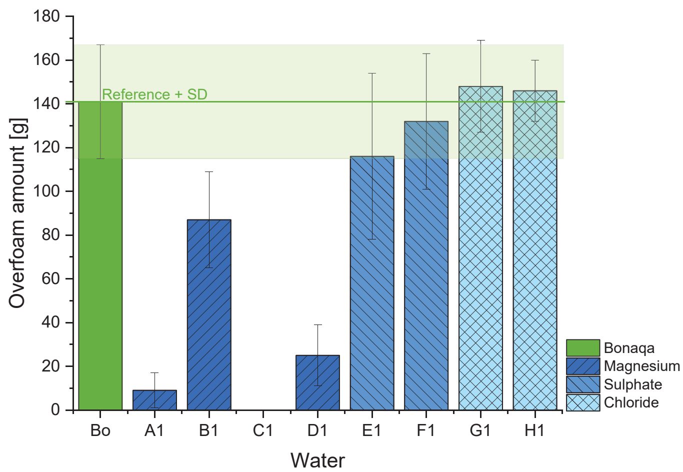

Fig. 1 Results of MCT in overfoam and its standard deviation, made with gushing positive malt and different waters samples - Bo: Reference Bonaqa®; A1 – D1: high magnesium content; E1: Sodium sulphate; F1: Calcium sulphate; G1: Sodium chloride; H1: Calcium chloride (n = 3)

Table 1 Ion composition of used test water. Bonaqa® water is identified as 'Bo'. Analysed ion content are indicated by (*), calculated values by (•)

Water	Ca^{2+} [mg/L]	K^{+} [mg/L]	Mg^{2+} [mg/L]	Na^{+} [mg/L]	Cl^{-} [mg/L]	SO_4^{2-} [mg/L]	HCO_3^{-} [mg/L]	ΣIons [mg/L]
Bo *	66	1.8	25.9	160	250	0.1	372	876
A1 •	2	2	89	2	260	11	0	366
B1 •	57	56	17	57	100	10	100	397
C1 *	109	124	77	144	279	316	250	1299
D1 •	2	2	75	2	11	260	0	352
E1 •	0	2	2	124	8	260	0	396
F1 •	110	–	–	–	8	260	0	378
G1 •	–	–	–	172	260	8	–	440
H1 •	150	–	–	–	260	8	–	418
A2 •	53	–	–	–	50	59	6	168
B2 •	102	–	–	–	100	119	6	327
C2 •	201	–	–	–	200	237	6	644
D2 •	–	104	–	–	50	50	6	210
E2 •	–	200	–	–	100	100	6	406
F2 •	–	391	–	–	200	200	6	797
G2 •	–	–	32	–	50	50	6	138
H2 •	–	–	62	–	100	100	6	268
I2 •	–	–	92	–	200	200	6	498
J2 •	–	–	–	65	50	50	6	171
K2 •	–	–	–	122	100	100	6	328
L2 •	–	–	–	234	200	200	6	640
Ak •		100			91			191
Bk •		200			181			381
Ck •		300			272			572
Dk •		100				123		223
Ek •		200				246		446
Fk •		300				369		669
Am •			25		73	–		98
Bm •			50		146	–		196
Cm •			100		292	–		392
Dm •			25		–	99		124
Em •			50		–	198		248
Fm •			100		–	395		495

The waters containing magnesium (A1 – D1) showed a reduced overfoam amount. In particular, waters A1 (Mg^{2+}_{A1} = 89 mg/L), C1 (Mg^{2+}_{C1} = 77 mg/L) and D1 (Mg^{2+}_{D1} = 75 mg/L) showed noticeable low overfoaming (0 to 25 g). Water C1 had, besides a high magnesium concentration, a total ion concentration of 1274 mg/L and showed no overfoaming in MCT. Similar results with high total ion concentration were observed by *Geißinger* et al. [18]. The water B1 also showed an OF reduction compared with the reference. However, the magnesium content was lower (Mg^{2+}_{B1} = 17 mg/L) and the OF decrease was less expressive.

In the case of waters identified in Figure 1 as sulphate, they have low (E1) or no (F1) magnesium content and showed just a slight decrease in OF (OF_{E1} = 116 ± 38 g; OF_{F1} = 132 ± 31 g) compared to the reference sample. In comparison, OF increased for waters containing chloride salts and no magnesium (G1 and H1: OF_{G1} = 148 ± 21 g; OF_{H1} = 146 ± 14 g). There was a deviation for water A1 (containing chloride) and D1 (containing sulphate). Water A1 was mineralised with mostly magnesium chloride and water D1 with magnesium sulphate. Water A1 showed a lower OF than water D1 in the tests, despite similar anion concentrations. However, the differences in OF between these waters are not statistically significant. The slightly higher magnesium concentration of water A1 may have a greater influence on the gushing behaviour than the present anions.

3.1 Single Cations

The following waters are composed by just one cation and chloride and sulphate anions respectively in similar contents. For these ions, their content were increased in three steps, whereas the concentration of hydrogen carbonate was kept constant at 6 mg/L in all samples. Figure 2 shows the results of gushing tests with waters containing single cations of calcium, potassium, magnesium and sodium in three ascending concentrations each.

The overfoam amount of these samples was lower than the reference (OF_{Bo} = 141 g ± 26 g). It is noticeable that in all analysed samples with the higher ion concentration (C2 = 201 mg/L Ca^{2+}; F2 = 391 mg/L K^{+}; I2 = 92 mg/L Mg^{2+}; L2 = 234 mg/L Na^{+}) an OF reduction occurred. However, in the case of waters containing K^{+} or Mg^{2+} a significant OF reduction was detected in all three concentrations (104, 200, 391 mg/L K^{+}; 32, 62, 92 mg/L Mg^{2+}). For potassium, a slight increase in OF can be observed at the highest concentration.

The lowest OF was observed at mean magnesium concentration (62 mg/L) and for concentrations of sulphate, chloride and hydrogen carbonate ions of 100 mg/L, 100 mg/L and 6 mg/L respectively as well as for a total ion concentration of 262 mg/L. (H2). If the ion concentration rises again (I2), the OF also increases. Furthermore, the decreased effect on OF is achieved with lower magnesium than potassium concentration. The OFs were higher if a single anion, chloride or sulphate, was present (see Fig. 3 and Fig. 4).

3.1.1 Investigation of potassium and magnesium ions with chloride or sulphate ions as accompanying anion

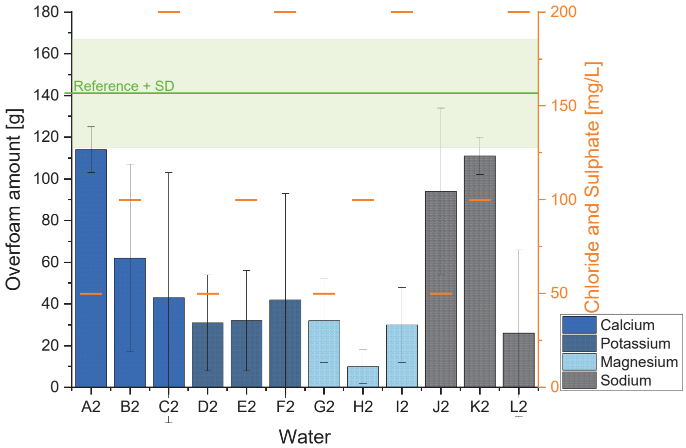

Fig. 2 Results of MCT in overfoam and its standard deviation, made with gushing positive malt and different waters with one cation and three anions (chloride, sulphate and hydrogen carbonate) (n = 3)

In the following, the influence of single cations with different anions on the gushing behaviour was investigated. For this purpose, potassium and magnesium ions were used as individual cations with chloride or sulphate ions in water in MCT in order to characterise their effects on gushing behaviour. Here, similar concentrations for chloride and sulphate should be achieved, that lead to different cation concentration for both cations. The overfoaming from MCT with waters containing potassium are shown in figure 3.

In the presence of individual ions, potassium ion as cation and chloride or sulphate ion as anion, the amount of overfoaming on MCT is lower than the OF with Bonaqa® water with gushing malt. With the increased ion content, the overfoam amount was initially raised, and then fell again. The lowest OF from potassium samples was measured with the highest sulphate concentration (OF_{Fk} = 64 ± 18 g; [SO_4^{2-}] = 369 mg/L), while the second lower OF can be seen with the lowest chloride content (OF_{Ak} = 83 ± 26 g; [Cl^-] = 91 mg/L). These results support the hypothesis that sulphate ions may have a gushing-reducing effect on the gushing behaviour in the MCT. However, a significantly lower OF (31 ± 23 g to 42 ± 56 g) could be achieved with potassium ions in the presence of several anions (Fig. 2). This indicates a reducing effect of the potassium ion on the gushing behaviour. Probably these ions change the surface tension by reacting with substances placed at liquid-gas surface. This should be principally analysed in the future.

Next, the influence of magnesium ions in combination with chloride or sulphate ions was investigated. The results are shown in figure 4.

The gushing behaviour of the magnesium-containing waters changed depending on the content of dissolved anions. The OF for individual anions was lower than the OF of Bonaqa® water with the same malt.

There is no significant difference in the OF between the waters Am–Fm. Already at lower concentration of magnesium (25 mg/L) and chloride (73 mg/L) a lower OF (57 ± 14 g) was obtained (Am). The highest OF (110 ± 8 g) of this series was measured at the lowest magnesium and sulphate concentrations. At a concentration of 50 mg/L Mg^{2+} there is a higher OF with Cl^- ions (80 ± 21 g) and a lower OF with SO_4^{2-} ions (59 ± 36 g) as accompanying anion. The results show a gushing-reducing effect of the magnesium ions on the gushing behaviour in the MCT, in the presence of chloride ions and in presence of sulphate ions too.

It is also evident that, as already found in the potassium test series, magnesium also showed a stronger gushing-reducing effect in combination with several anions: chloride, sulphate and hydrogen carbonate. The OF from waters containing magnesium (G2 to I2) was 10 to 32 g, whereas in the case of Am to Fm (Fig. 4) it was 57 to 110 g. Overall, the waters containing magnesium ions demonstrated the lowest OF between all the samples.

4 Conclusion/Summary

The results showed that the overfoam amount in gushing test could be influenced by the ion composition and their concentration in the used water. A gushing-reducing effect could be seen for potassium and magnesium ions in combinations with several anions – sulphate, chloride in similar concentrations and hydrogen carbonate

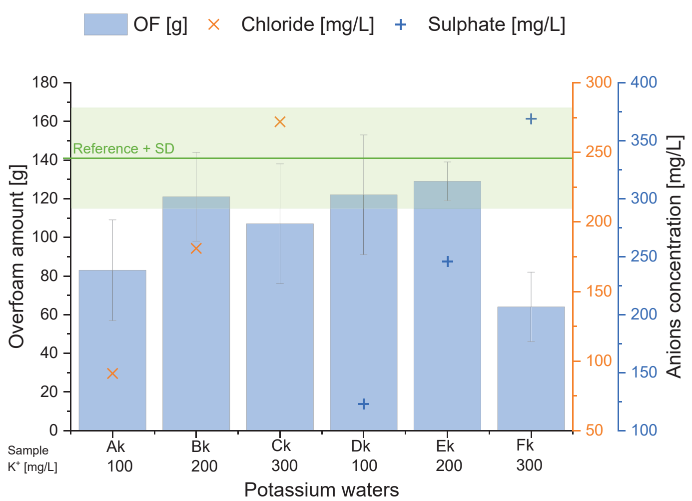

Fig. 3 Result of MCT using potassium containing waters (n = 3)

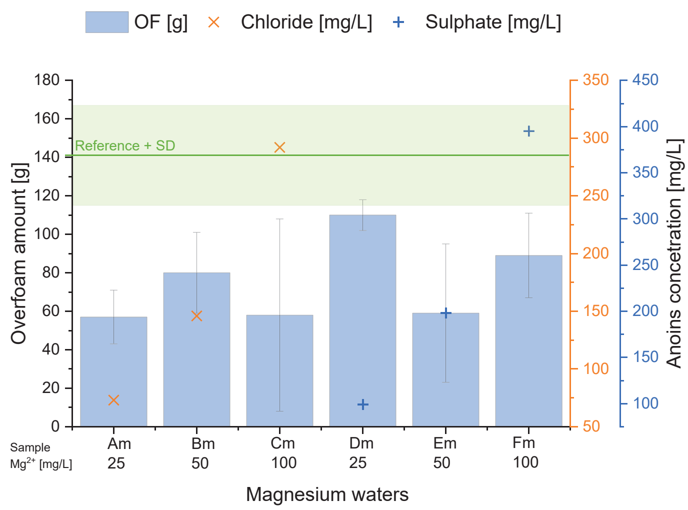

Fig. 4 Results of MCT using magnesium containing waters (n = 3)

in low concentration. Magnesium in particular causes a significant reduction in OF already in concentrations of 30 to 60 mg/L, which are lower than the concentrations of the others analysed cations.

The findings obtained provide another piece of the puzzle to solve the problem of gushing: More important than the individual ions is the effect of the composition of ions present in the water. Further research is desirable to determine to what extent the composition of the ion matrix in terms of cations influences the gushing behaviour of a malt in MCT. One possible approach would be whether the simultaneous presence of potassium and magnesium ions, together with chloride and sulphate ions, will reduce the OF. It is still unclear whether there is a minimum and/or maximum limit of ion concentration at which gushing is reduced. Further research is also desirable to verify the results for other gushing malts as well.

In the brewing process there is an intake of ions from the malt, with the potassium and magnesium content in final beer to be about 550 mg/L and 100 mg/L respectively [29]. These may already have a gushing-prevent effect in beer. Since ions react in the brewing process, they are also able to react with substances placed in the surface liquid-gas and change the surface tension that leads to the reduction of overfoam amount by gushing beer

Although, the ions influence the gushing behaviour in MCT, the change of brewing water has to be analysed for each case, taking into account the tendency of beer gushing in each brewery and the possible changes in the process and in the beer's final taste.

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