



## Evaluation of the sulfate/chloride ratio in craft beer production and its sensory impacts

### Avaliação da relação sulfato/cloreto na produção de cerveja artesanal e seus impactos sensoriais

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#### ABSTRACT

The craft beer market has stands out in the national and international economic scenario. In this way, the search for news and innovation has increased exponentially, arousing the curiosity of many connoisseurs and specialists in the area. In this context, the present work aims to analyze the sensory influence related to changes in the proportion between sulfate and chloride ions present in the water used to produce artisanal beer. For this purpose, three samples of a German Kölsch style beer were produced, with a neutral profile, with different relationships between these ions. Thus, through sensory analysis tests, the influence of these ions on the final product was verified, in addition to its acceptability index. The result of this analysis showed that there was a considerable difference between the sensory perceptions of malt bitterness and sweetness related to the concentration of the highlighted ionic species, demonstrating that such modifications can contribute satisfactorily to the quality and acceptance rate of the product. However, these correlations must be planned according to the style of beer to be produced.

#### RESUMO

O mercado das cervejas artesanais tem se destacado no cenário econômico nacional e internacional. Desse modo, tem-se aumentado exponencialmente a busca por novidades e inovação, despertando a curiosidade de muitos apreciadores e especialistas da área. Nesse contexto, o presente trabalho tem como objetivo analisar a influência sensorial relacionada às alterações na proporção entre os íons sulfato e cloreto presentes na água de produção da cerveja artesanal. Para tanto, produziu-se três amostras de uma cerveja do estilo alemão Kölsch, de perfil neutro, com relações distintas entre esses íons. Assim, por meio de testes de análise sensorial, verificou-se a influência dos referidos íons no produto final, além seu índice de aceitabilidade. O resultado dessa investigação mostrou que houve uma diferença considerável entre as percepções sensoriais de amargor e doce do malte relacionados à concentração das espécies iônicas em destaque, demonstrando que tais modificações podem contribuir satisfatoriamente na qualidade e no índice de aceitação do produto. Entretanto, essas correlações devem ser planejadas de acordo com o estilo de cerveja a ser produzido.

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## Introduction

Beer is one of the oldest and most consumed drinks in the world, with its production playing a relevant role in the world economy. In Brazil, the number of beer producing establishments registered with the Ministry of Agriculture, Livestock and Supply (MAPA) reached the mark of 1,549 breweries in 2021, which represents an increase of 12.0% compared to the previous year, when there were 1,383 registered breweries, thus demonstrating a full rise in this industrial segment (Brasil, 2021, p. 23).

In terms of product, according to Law No. 8,918 (Brazil, 2019), which provides for the standardization, classification, registration, inspection and production of beverages, beer is characterized as a beverage obtained from the alcoholic fermentation of brewers must Coming from barley malt and drinking water, through the action of yeast, with the addition of hops. In this sense, for good quality in the manufacturing process, all stages must be rigorously monitored, from the malting process to the final product.

In relation to their variety, beers are distinguished by several aspects, such as: color, alcohol content, type of fermentation and bitterness index. It is believed, therefore, that at present there are more than 20 thousand types of beers worldwide (Venturini, 2005). Therefore, what differentiates such variations are, most of the time, subtleties in the manufacturing process, such as different grain cooking times and temperatures (mash), fermentation/maturation and the use of some ingredients in addition to the basic inputs (Brasil, 2019).

Among the fundamental ingredients, production water stands out for having a direct influence on the flavor, foam, sensorial stability and color of the beer. The main minerals present in water that effectively affect sensory characteristics are: calcium, chloride, sulfate, carbonates and bicarbonates, sodium, magnesium and zinc (Souto, Pagorari & Solgon, 2021).

For centuries, breweries that extracted their production water from wells or springs consolidated entire regions as major beer producers, giving them characteristics related to the quality and composition of this water source. Thus, the city of Burton, in England, is an example, whose water used in manufacturing processes had high hardness due to the high content of calcium sulfate, resulting in a production center for bitter, strong and clear beers. In the case of London and Munich, as they had more alkaline water, darker beers were obtained with a smoother and lighter flavor, like a brown ale, or lightly hopped like a lager (Priest & Stewart, 2006, p. 10).

With technological development, it is currently possible to modify the salt profile present in water according to each style of beer. However, it is essential to have in-depth knowledge of how each salt contributes sensorially to the beer being produced, as the way in which each salt works varies significantly for each recipe.

According to the context,  $\text{CaSO}_4$ , known as gypsum or gypsum, is used in brewing water with the purpose of increasing the hardness of the water (consequence of calcium), in addition

to providing a better sensation of hop bitterness (consequence of sulfate) . Thus, high concentrations of sulfate favor the production of beers with high levels of bitterness, such as Indian Pale Ale (IPA) or English bitters. However, in conditions of high concentration, these ions can bring astringency and excessive sulfur notes (Hornink & Galembeck, 2019, p. 175).

Calcium, available in the aforementioned salt, is essential for regulating the enzyme  $\alpha$ -amylase, whose function is to hydrolyze the glycosidic bonds present in the structure of substrates such as glycogen and starch. In the beer production process, depending on the style you want to obtain, a calcium concentration of up to 200 ppm is suggested. (Siqueira, Bolini & Macedo, 2008, p. 497).

In the case of  $\text{CaCl}_2$ , it helps to highlight the flavor of the malt (sweetness), in addition to helping to increase water hardness and pH. Typically, it is recommended to use between 50 and 200 ppm of calcium and around 0 to 200 ppm of chloride. Unlike sulfates, a high load of chlorides is not suitable for the production of bitter beers, being more suitable for styles that have a high amount of malts rich in non-fermentable sugars, such as Porters and Stouts (Hornink & Galembeck, 2019, p 59).

There is also a range of salts with great sensorial influence on beer, such as sodium chloride ( $\text{NaCl}$ ), Calcium Carbonate ( $\text{CaCO}_3$ ), Magnesium Sulfate ( $\text{MgSO}_4$ ), Sodium Bicarbonate ( $\text{NaHCO}_3$ ) and Zinc Sulfate ( $\text{ZnSO}_4$ ), however, emphasis will only be placed on  $\text{CaCl}_2$  and  $\text{CaSO}_4$ , the objects of this discussion.

Therefore, the main objective of this work is to analyze the sensorial influence related to changes in the proportion between sulfate and chloride ions present in the production water of craft beer.

## **Material and methods**

To obtain the featured product, a recipe was developed based on the German style Kölsch, originating from the city of Cologne. According to the definitions of the Beer Judge Certification Program (BJCP) (Strong, 2021, p. 9), the style features a pale colored, high fermentation beer, obtained from German yeast (ALE), medium bitterness derived from noble hops of German or Czech origin, neutral flavor, similar to a German or Czech lager. The Brewers Association Beer Styles Guidelines guide (Papazian, 2022, p. 20) was also consulted as a complementary reference, seeking to fully understand the style produced. The technical parameters for preparing the recipe were obtained by BeerSmith<sup>®</sup> Software (Smith, 2023). Table 1 provides the relationship between the parameters described in the BJCP and the recipe developed.

**Table 1.**

Relationship between the developed recipe and the BJCP technical reference.

<b>Technical attributes</b>	<b>BJCP Parameters Kölsch (5B)</b>	<b>Parameters of the produced recipe</b>
Bitterness (IBU)	18 - 30	20
Color (SRM)	3,5 - 5	4,5
Initial Density (g/mL)	1,044 - 1,050	1,047
Final Density (g/mL)	1,011 - 1,017	1,011
Alcohol Content (ABV%)	4,4 - 5,2	4,8

\*IBU: International Bitterness Unit

\* SRM: Standard Reference Method – American parameter

Origin: Own Author

Regarding the equipment, density values were established using a glass densimeter with a scale of 0.980/1.100, manufacturer INCOTERM. The color and bitterness parameters were simulated by the BeerSmith® software, taking as a reference the characteristics and quantities of the selected malts and hops. The recipe was executed in a craft brewery located in the metropolitan region of Fortaleza (CE), using equipment measured for a maximum production of 50 liters.

Water, an essential element in the production of beer, is the ingredient with the largest composition, therefore, it is essential that no type of water is used. This ingredient must be free of microorganisms, odorless, and without the residual taste of chemical treatments. The balance of mineral salts present in this ingredient can also profoundly influence the sensory experience, highlighting aromas and flavors. Otherwise, their imbalance can destabilize and bring unpleasant flavors such as salty, residual bitterness, astringency, among others (Palmer, 2013).

Thus, to execute the recipe, four gallons of commercial mineral water of an unknown brand were used, containing five liters each. Therefore, according to the information provided by the manufacturer, its composition is represented by Table 2.

Table 2. Composition of the mineral water used

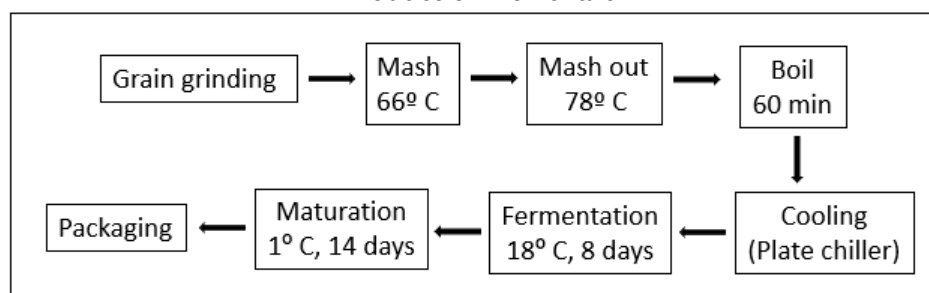
<b>COMPONENT</b>	<b>AMOUNT (mg/L)</b>
calcium	9,3
Magnesium	13,8
Potassium	0,53
Sulfate	4,5
Chloride	109,0
Bicarbonate	153,4
Sodium	82,1
	7,1

Origin: Manufacturer of the brand of mineral water used

At the end of the process, 13 liters of beer were produced.

Therefore, the manufacturing process followed the steps described in the production flowchart below:

**Figure 1.**  
Production flowchart



Origin: Own author

To obtain the evaluated samples, the total volume was initially divided into three batches of 4.3 liters, considering 0.7 L for the head space to be used in fermentation. In this way, the samples were fermented in the same gallons that contained the total water, so that each one received a specific salt correction.

All additions were calculated using BeerSmith® software (Smith, 2023). pH measurements were obtained from a MY140 MYLABOR pH meter, with a measurement sensitivity of 0.01 and a working temperature of 0° to 100°C. The values referring to the additions made, the final concentrations and the pH of the solution after salt modification are described in Table 3:

**Table 3.**  
Additions of Cl<sup>-</sup> and SO<sub>4</sub><sup>2-</sup> ions in each sample produced.

	<b>Batch 01</b> Cl <sup>-</sup> /SO <sub>4</sub> <sup>2-</sup> (1:2,3)	<b>Batch 02</b> Cl <sup>-</sup> /SO <sub>4</sub> <sup>2-</sup> (1:1)	<b>Batch 03</b> Cl <sup>-</sup> /SO <sub>4</sub> <sup>2-</sup> (3:1)
<b>Addition of salts</b>	1,9 g de CaSO <sub>4</sub>	0,8 de CaSO <sub>4</sub>	0,6 de CaCl <sub>2</sub>
<b>Concentration after addition</b>	251 mg/L (SO <sub>4</sub> <sup>2-</sup> )	109 mg/L (Cl <sup>-</sup> e SO <sub>4</sub> <sup>2-</sup> )	254,8 mg/L (Cl <sup>-</sup> )
<b>Final product pH</b>	4,5	4,65	4,4

Origin: Own author

Thus, for the first batch, 1.9 g of CaSO<sub>4</sub> was added in 4.3 liters. Under these conditions, a sulfate content of 251 mg/mL is obtained (sulfate/chloride ratio: 2.3: 1). According to Tozetto (2017), the acceptable limit of sulfate is between 50 ppm and 250 mg/L, favoring beers with a bitter profile at their upper limit. In this case, it was decided not to exceed this limit, as excess sulfate ions can bring unpleasant astringency and bitterness to the beer. According to Palmer (2013), the sulfate/chloride ratio in beer production is only relevant when these ions are found in quantities between 50 and 250 mg/L, becoming insignificant at lower concentrations.

In the second batch, 0.8 g of CaSO<sub>4</sub> was added in 4.3 liters. Under these conditions, the sulfate/chloride ratio remains balanced (1:1). As the water used already naturally had a

high chloride content, only a small addition of  $\text{CaCl}_2$  was added, thus obtaining quantitative balance between these two ions.

In the third batch, 0.6 g of  $\text{CaSO}_4$  and 1.3 g of  $\text{CaCl}_2$  were added, in order to obtain a concentration of 254.8 mg/L of chloride ions (sulfate/chloride ratio: 1: 3). According to Tozetto (2017), to favor a malty profile, the amount of chlorides must reach the limit of 250 ppm, which requires a very robust malt base to support this addition without bringing salty notes. Above this, sensory sensation can be compromised. At the end of the process, three distinct samples were obtained, with different chloride/sulfate ratios.

Following the events, a sensory test was administered to 40 volunteers, of both sexes, aged at least 18 years and who would not drive a vehicle for the next four hours.

The method used for sensory evaluation was of the “paired comparison test” type. According to Meilgaard (1987), the objective of the test is to know whether a sample presents a certain sensory attribute in greater intensity than the other sample. It is worth mentioning that this type of test has a directional characteristic, as it draws the taster's attention to a certain sensory profile (sweetness, acidity, etc.).

At this stage, the participant group was divided into two subgroups: untrained participants (60% - 24 people) and participants with sensory experience in craft beers (40% - 16 people). The group of those not trained was basically composed of university students studying Chemical Engineering at a Higher Education Institution, located in the city of Fortaleza – CE. The second group included: beer production professionals, sommeliers and members of the Ceará Craft Brewers Association – ACERVA.

The method used to collect and evaluate data was quantitative descriptive analysis. According to Stone and Sidel (2004), this is the most used sensory description technique in the food area, as it allows the survey, description and quantification of the sensory attributes detectable in the product, using evaluators with varied training profiles.

This methodology involves three fundamental steps: 1. surveying attributes and familiarizing evaluators with the products; 2. the definition, in consensus with the team of evaluators, of the descriptor terms and the establishment of references that will serve as intensity standards (minimum and maximum) for each attribute; 3. evaluation of samples normally using an unstructured 9-point scale to quantify the intensity of sensory attributes (Alcantara & Freitas-Sá, 2018).

When applying the tests, each volunteer received three samples containing 100 mL each, in transparent plastic cups identified by codes. To prevent participants from communicating in a way that could cause any type of sensory influence during the test, assessments were made individually.

To capture the responses, an electronic form created from Google Forms® was used, containing objective questions presented on a 9-point hedonic scale, with 1 being extremely disliked and 9 being extremely liked. In this way, the parameters of color, flavor, aroma,

appearance, global acceptance, acceptability index (%) and presence of off flavors were evaluated (Minin, 2009).

To obtain acceptability indices (AI%), the equation proposed by Dutcosky (1996) was adopted:

$$\text{Product Acceptability Index (AI\%)} = A \times \frac{100}{B} \quad \text{Equation 01}$$

Where A represents the numerical value of the average score obtained by the evaluated attribute and B the maximum score given to that attribute. According to Dutcosky (1996), for a product to be considered accepted, it must obtain an Acceptability Index of at least 70%.

The questions applied in the research can be accessed through the QR Code shown in Figure 2.

**Figure 02:**

QR code with the path (link) to the survey questions.



Origin: Own author

As described by Creswel (2007), the quantitative data treatment process occurred based on descriptive statistical analyses, aiming at the logical-rational organization of data distribution. Thus, the data collected was carefully tabulated and analyzed according to the pre-established objectives of the investigation.

It is worth noting that the participants in this research agreed to the terms of free and informed consent, which is also included in the QR core represented in Figure 02. Thus, all the steps inherent to the research were detailed in the document, in addition to emphasizing the importance of their holdings.

## Results and discussion

Based on the data collected, the samples were evaluated according to their sensory characteristics acquired with changes in the salt composition of the production water. Therefore, these parameters can be analyzed from Table 4:

**Table 4.**

Data collected from sensory tests

<b>Parameters analyzed</b>	<b>Sample 01 SO<sub>4</sub><sup>2-</sup>/Cl<sup>-</sup> (2.3:1)</b>	<b>Sample 02 SO<sub>4</sub><sup>2-</sup>/Cl<sup>-</sup> (1:1)</b>	<b>Sample 03 SO<sub>4</sub><sup>2-</sup>/Cl<sup>-</sup> (1:3)</b>
Perception of bitterness	47.5% (19)	37.5% (15)	15.0% (6)
Sweetness perception	37.5% (15)	30.0% (12)	32.5% (13)
Sensory preference	55.0% (22)	17.5% (7)	27.5% (11)

Origin: Own author

Through objective questions, participants were asked which samples performed best in terms of bitterness perception, malt sweetness and general sensory experience/preference. Therefore, sample 01 was chosen as the one that provided them with the best sensory experience. The hypothesis created is that the salt profile of the sample goes beyond the recommended limit of sulfate content, favoring the bitterness parameter.

It is believed, therefore, that in the minds of admirers of craft beers, a characteristic closely related to this product is the evident bitterness, therefore, the influence of sulfate in high quantities may have directly influenced the result. In the case of sample 03, it presented a relevant disadvantage in relation to sample 01, possibly due to the salty notes added by chloride in high concentrations.

According to Oliveira (2019), the creation of recipes with very high chloride levels, for beers with a neutral profile and low gravity, add brackish notes to the final product, resulting in a sensorial imbalance.

In this context, as reported by Tozetto (2017), a very dense malt base would be needed to enhance the sweetness without presenting sensory imbalances related to chloride ions. Therefore, for the Kölsch style, the focus on malt sweetness was not relevant, although for styles that use darker malts, with a high content of non-fermentable sugars, this change is more coherent.

According to Salimbeni (2016), the addition of sulfate highlights the sensation of "crispness" or dryness in the hop bitterness, making its flavor less astringent, improving the flavor and quality of the bitter character.

During the production process of the object of this investigation, the addition of CaSO<sub>4</sub> decreased the pH of the must, as demonstrated in Table 03 above, however, such additions did not contribute to the formation of undesirable by-products, off flavors. According to Palmer (2013), the average pH of a finished beer varies between 4.0 and 4.6 (lagers) and 4.0 and 4.4 (ales). The author also emphasizes that changing the pH of the environment, when done correctly, contributes to the breakdown of starch into fermentable sugars and the process of protein degradation, in addition to helping with filtering and the formation of the protein body that precipitates in the center of the mash tun at the end of the boiling process.

According to Barth (2013), by drastically reducing the initial pH value of the production water, which has an average pH value between 6.5 and 7.0, characteristics such as metallic



flavor and /or acidic (pH<3.7). Therefore, when exceeding the recommended limits for adding salts, special attention must be paid to the pH of the production water.

Regarding additions of CaSO<sub>4</sub>, when it is added in large quantities, it can also favor the production of sulfur compounds during fermentation, negatively influencing the sensorial quality of the product (Salimbeni, 2016).

As reported by SENAI (2014), to produce beers with a bitter character, such as India Pale Ale or American Pale Ale, a sulfate/chloride ratio of up to 5:1 is suggested. In this way, the flavor, aroma and bitterness of the hops are highlighted. If the addition of this single salt (CaSO<sub>4</sub>) is not sufficient to achieve the desired profile in relation to the other ions (Mg<sup>2+</sup>, Cl<sup>-</sup>, Ca<sup>2+</sup>, HCO<sub>3</sub><sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, Na<sup>+</sup>), it is necessary to combine other salts. Thus, calcium carbonate, for example, can be used to introduce temporary hardness in water and raise the pH of the mash in dark beers. Calcium chloride, to introduce permanent hardness and enhance the sweetness of the beer, Sodium bicarbonate (NaHCO<sub>3</sub>) to correct pH, making the medium more alkaline, among others.

According to Salimbeni (2016), for the production of dark beers, for example, Stouts, Bocks and Porter, water with moderate hardness is recommended. Therefore, the salts commonly used to increase the hardness of production water are CaCl<sub>2</sub>, CaSO<sub>4</sub> and MgSO<sub>4</sub>. Thus, the flavor that will be incorporated into the beer is described by the author as “dry”, slightly astringent and sparkling. Therefore, an ideal ratio between these ions cannot extrapolate a sulfate/chloride ratio of 1:2 (Salimbeni, 2016).

As a way of evaluating the general performance of the samples, the product acceptability index (AI%) was measured, described in Table 5.

**Table 5.**  
Acceptability index of the evaluated samples.

<b>Parameters evaluated</b>	<b>Average grades</b>	<b>Acceptability index (%)</b>
Appearance	8.2 ± 0,11	91.1
Color	8.5 ± 0,23	95.5
Aroma	7.6 ± 0,29	87.3
Flavor	7.5 ± 0,33	85.2
Texture	7.2 ± 0,12	83.7
Global Assessment	7.7 ± 0,38	86.5

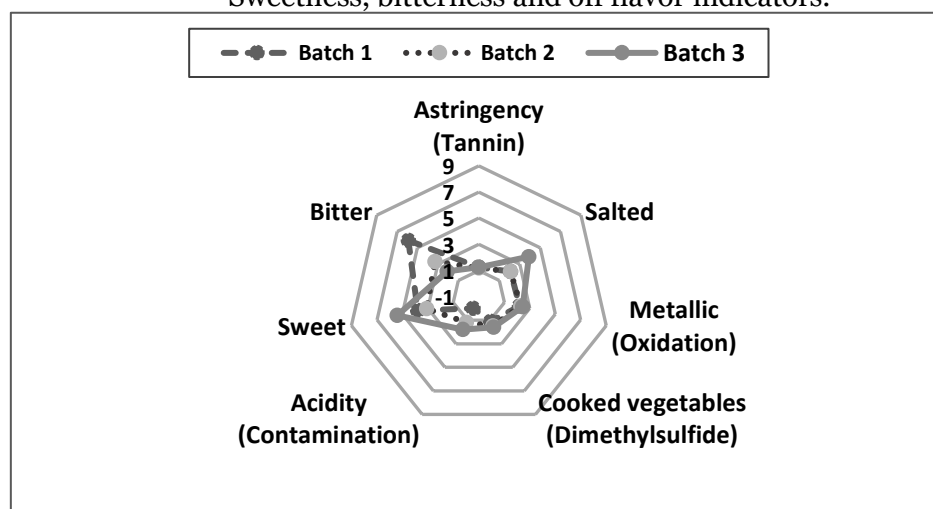
Origin: Own author

Based on the tabulated data, the samples evaluated showed good average acceptance, as the AI% values were above 80%. This way, it can be seen that the recipe was well executed, even though batch O3 had a residual salty flavor. It is also observed that factors related to flavor and texture, as previously discussed, were the most affected in the sensory evaluation, possibly reflecting a small decrease in AI%. However, the product, when analyzed as a whole, received good acceptance from evaluators, even those more experienced.

According to Axcell & Torline (1998), in the AI% test it is observed that non-experienced consumers do not have the knowledge or special attention to detect differences in specific attributes, which are easily detected by a trained team. Thus, according to Araújo, Silva & Minin (2003), samples may be more or less bitter or sweet in relation to others, however evaluators may not consider them as negative enough attributes to interfere with the degree of acceptance. In any case, the evaluations carried out by the experienced evaluators were in line with those defined by the non-experienced evaluator.

Aiming to evaluate the quality of the product obtained, the sensorial perception of some parameters indicating errors or production defects, also known as off flavors, was analyzed. Thus, from the graph described in Figure 2, it can be seen that the evaluated product did not present characteristics beyond those expected for the style produced.

**Figure 2:**  
Sweetness, bitterness and off flavor indicators.



Origin: Own author

As shown in Figure 2, for the malt sweetness and bitterness characteristics, the values obtained in the tests corroborate the changes made in the composition of the sulfate and chloride ions. Thus, it is observed that batch 01, whose sulfate ion concentrations were extrapolated to highlight the bitter taste, stood out in this parameter.

According to Brunelli (2014), the perception of bitterness in a beer is mainly related to the use of hops. The use of this ingredient as a bittering agent is well established and widely disseminated in beer circles. This characteristic is related to the presence of substances called alpha-acids, present in hop flowers and which isomerize during the boiling stage, giving rise to the desirable bitter flavor in beer. While hops are responsible for bitterness, they can also provide aromas and other flavors from substances popularly known as essential oils, belonging to the terpene class. (Felipe & Bicas, 2017).

However, it is not common, nor desirable, to use heavily toasted malts to achieve a certain level of bitterness. In fact, most of the time this type of bitterness from malt is never well received in a beer (Smith, 2023).

In the case of the beer evaluated, the batch whose addition of CaSO<sub>4</sub> was emphasized (batch 01) demonstrated that the bitterness characteristic can be highlighted without changing the type or quantity of malt or hops. However, it is necessary to be cautious and have technical knowledge of what you want to obtain, otherwise the bitterness obtained may be unbalanced.

For batch 03, which had its CaCl<sub>2</sub> concentration extrapolated, it is clear that the sweetness characteristics of the malt were emphasized. However, in the “salty” criterion, the batch presented an average score of 3.9, which denotes that this characteristic was present. This demonstrates that a base of malts rich in non-fermentable sugars is essential for high concentrations of chloride to act in balance with the recipe.

According to Salimbeni (2016), a strong addition of Cl<sup>-</sup> ions positively contributes to the balance between sweetness and bitterness in beers with a high amount of non-fermentable sugars, however, such concentration should not exceed a sulfate/chloride ratio of 1:2, if otherwise, salty notes can unbalance the desired sensory characteristics. This concentration must therefore not exceed the 100 ppm (mg. L<sup>-1</sup>) mark.

Finally, the execution of the recipe stands out, especially in sample 01, which was balanced, even in the face of extreme modifications, thus representing a product with good sensorial stability and commercial potential. In the case of sample 03, it is necessary to rethink the recipe in terms of quantities of special malts or adequacy of the sulfate/chloride ratio.

### **Final considerations**

Given the above, the present investigation demonstrates that the manipulation of salts in the craft beer manufacturing process contributed to the two samples that had their sulfate and chloride levels unbalanced (samples 1 and 3) obtaining a good acceptability rate in sensory evaluations. . Thus, the results presented demonstrated that the correct handling of brewing water, according to the characteristics of the recipe, is largely favorable for improving the quality of the final product, since, in this way, desirable characteristics can be highlighted.

In this sense, beers that naturally have malt sweetness as a focus, for example, porters and stouts, when produced with an eye on the imbalance that benefits the concentration of chloride ions, will possibly present greater complexity and balance in the final product. Also, naturally bitter beers such as India Pale Ale or English Bitter will have their bitterness characteristics favored through the creation of recipes that favor the concentration of sulfate ions in relation to chlorides.

Set the final evaluation of the product in light of the modifications made, the proposed recipe, especially from batch 01, presented itself with relevant commercialization potential.

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