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Blending of hard seltzer and other flavor intensive beverages inline vs. batch – with a focus to the flavor-losses

Batch vs. continuous – an often and widely discussed topic. Economic comparisons are the basis for decision-making for the optimal selection of the process or the consideration of investing in the development of continuous processes. In addition to the economic background, the process time, the ability to automate, the cleaning effort, the product losses and the maintenance effort - for the producers of flavor intensive products, the quality and the influence of the process on the product are in the foreground. This publication is intended to show the extent to which a batch process for mixing Hard Seltzer and other flavor-induced beverages with the batch-vessels in use for this purpose changes the products regarding their characteristic flavors. To be able to sufficiently verify this in addition to the theoretical models, the dependencies of the relative volatilities that have been developed in this context are included and presented.

Descriptors: volatility, hard seltzer, evaporation, flavor, activity coefficient, infinite dilution

1 Introduction

In many industries the question arises for a producer whether a continuous or a batch process is the more economical and product-friendly solution. In the brewery industry in particular, batch processes for mixing, blending, and producing mixed beer beverages are established processes. In the production and flavoring of Hard Seltzer, (carbonated) soft drinks (CSD), fruit juices and similar beverages, where flavors are in the foreground of the product characteristics and the organoleptic perception, the decision which kind of process is used, can be decisive for the success of the products. Regarding the loss of flavor, the focus has so far always been on the thermal product treatment and the filling process. Loss of flavor or changes of more than 5 % already lead to considerable criticism of the system.

The quantification of flavor reductions by means of gas chromatography as part of a process stage control (discharge of finished beverage vessels, before / after vacuum degassing, before / after filling process) also leads to considerable fluctuations. So far, these have been attributed to the high error tolerances in sampling (oxidation, losses, etc.) and the analysis, which does not lead to a clear statement with regard to the specifications to be complied with in the event of loss of flavor. Furthermore, values for the volatility of the key flavor limonene are missing for a proper thermodynamic quantification. Discrepancies now lead to the assumption that the batch processes in the upstream product mixing and emptying contribute much more negatively to the preservation of the flavors than previously assumed.

In the following, the associated emptying of the vessel and the associated change in the flavor profile after the individual components have been brought together and mixed with a carrier liquid (water) will be considered in a batch process.

1.1 Procedure for emptying the vessel and setting the thermal equilibrium

The process picture shown in figure 2 (see page 135), describes the process of emptying the vessel:

The vessel ***T*** to be emptied is usually pressurized with sterile air or an inert gas (CO_2 / N_2) at a constant pressure ***p***. During the emptying process, a pump draws off the liquid F with the flavor concentrations ***cAa*** (mass fractions) with decreasing liquid supply ***F*** and enlargement of the gas space ***G*** at the vessel outlet. To maintain the pressure ***p***, sterile air or inert gas must be feed in at the inlet via a control valve ***RV***. At the same time, vaporous flavor substances (quantities ***ΔmA***) and water vapor (quantity ***ΔmW***) escape from the liquid into room ***G***. The gas mixture **GM** formed in this consists of flavor vapor (mass ***MAG***), water vapor (mass ***MWG***) and air or inert gas (mass ***MIG***) together. The liquid ***F*** consists of flavor (mass ***MAF***), water (***MWF***) and other ingredients. A thermal equilibrium is established between the solution ***F*** and the gas mixture ***GM***. The flavor concentrations ***cAa*** result from the equilibrium state. Their comparison with the preparation data provides the result for the change in the flavor profile when the vessel is emptied [2, 6].

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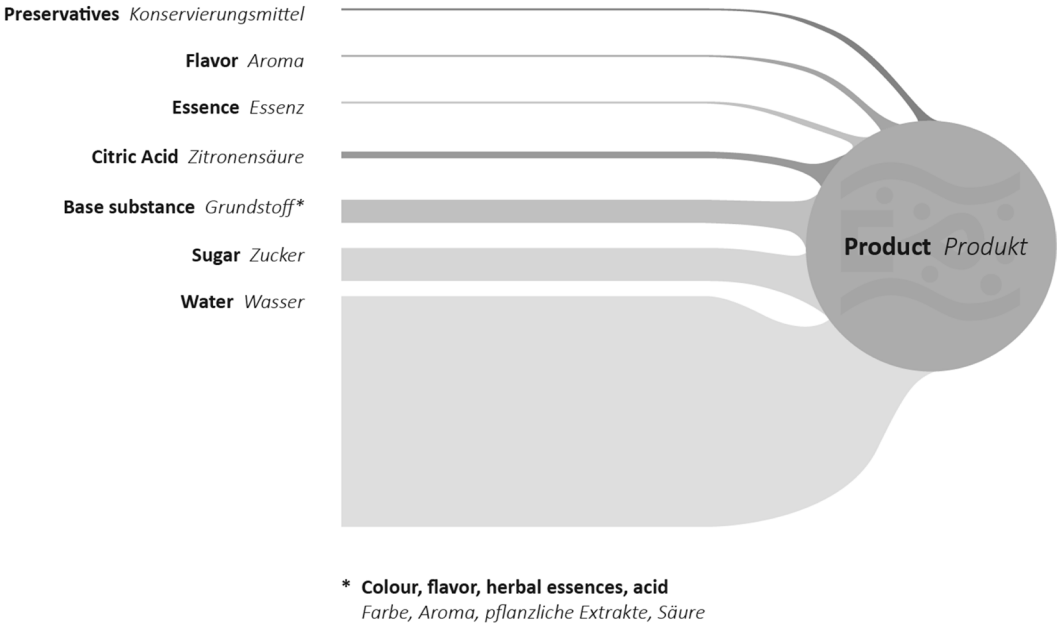

Fig. 1 Batch product blending of for this example 7 different components to the final product [11]

When calculating flavor losses, one of the most important parameters is the relative volatility of the corresponding key flavors in the beverage. In general terms, the relative volatility α defines which substance is more volatile - the solvent or the flavoring substance. For $\alpha < 1$ the substance is not very volatile for $\alpha > 1$ the substance is highly volatile, and the solvent will be depleted. In the literature either no values are described for these beverage flavors, or values can be found that were determined without taking into account the composition of the solvent or the temperature dependency. The following results and considerations should on the one hand provide the influence of the process selection (batch or inline) on the product quality, and on the other hand provide results for the calculation of the quantification of flavor losses.

In this publication, in order to clarify and standardize the various statements, the meaning of volatility is illustrated, thermodynamically defined and confirmed in the form of simple experiments, proven by literature data on flavoring substances typical of beverages (including benzaldehyde and limonene). The result is that in order to estimate the loss of flavor in the manufacturing process, a simple consideration of the relative volatilities, especially in concentration levels of infinite dilution, can lead to major errors.

To illustrate the problem, the matrix of the beverage must be considered. This is made up of various components. The main component of Hard Seltzer and blended beverages is undoubtedly water, and there are also ethanol, various sugars, and substances in water in a dissolved and undissolved state. In this publication, the flavoring substances and their behavior in water are of great importance. The influence of ethanol in the solution should also be considered. These flavorings are very different in themselves, but they all have in common that they are highly diluted in the main component water.

The following question arises: How is the behavior of the mixture to be described and calculated?

Part of the answer can be found in looking at the interactions that define the mixture: water molecules interact equally with one another. More precisely, all water molecules attract each other with a certain strength or, depending on their position, also repel each other. But there are no water molecules that attract or repel other water molecules more strongly, water molecules are indistinguishable here. These considerations also explain the surface tension of water. The interaction with air molecules is of a different nature and strength, so that the limiting water molecules experience an attraction downwards, towards the other water molecules. The lack of upward attraction results in surface tension. This can be seen very clearly with water droplets in weightlessness.

If there is a flavor molecule in water that interacts in the same way with water molecules, then an ideal mixture is present. Ideal because there is only one type of interaction in the entire liquid. This interaction is the same between the individual water molecules, it is the same between a flavoring substance and a second flavoring

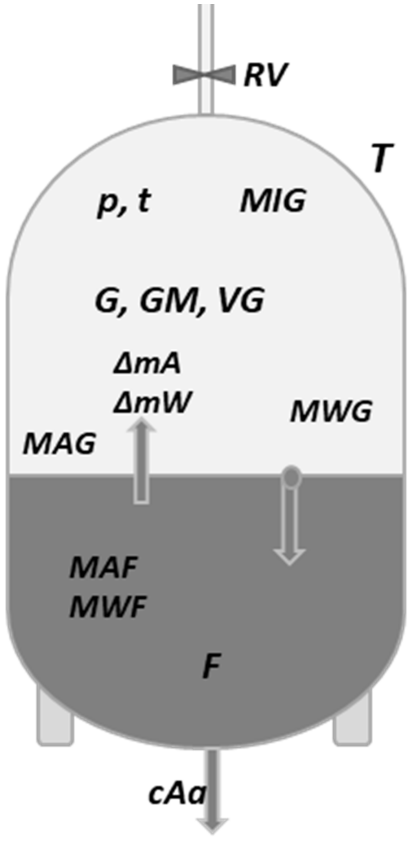

Fig. 2 Procedure for emptying the vessel and setting the thermal equilibrium [2, 6]

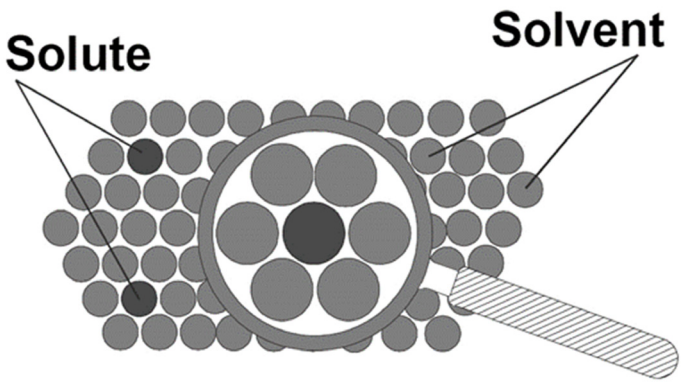

Fig. 3 Depiction of infinite dilution [3, 12]

substance molecule and it is the same between flavoring substance and water molecules. This ideal case is often described in the literature [8] with the benzene-toluene mixture. These are almost indefinitely soluble in one another.

However, a variety of flavorings are present in a flavored beverage. Therefore, the ideality described is the exception. This simplification does not apply to most flavoring substances and three different interactions occur in a mixture consisting of water and a certain flavoring substance. These consist in the interaction between water molecules, in the interaction of the flavor molecules and in the interaction of the flavor-water molecules. There is now a real mixture, which is much more complex to describe in terms of its thermodynamic behavior. If four components (water, ethanol, flavor 1, flavor 2) must be considered, the number of possible interactions increases rapidly. There are the four mentioned interactions and, in addition, the interactions between molecules of flavor 2, between water and flavor 2 molecules and between flavor 1 and flavor 2 molecules.

Despite the interactions, the Hard Seltzer, as just one example of many beverages, is fundamentally simplified here due to the high degree of dilution of the individual flavor molecules in the juice matrix. The flavor substances are so highly diluted that interactions between the molecules of a flavor substance (flavor substance 1) and between other, different flavor substances (flavor substance 2, ...) hardly take place. Thus, the interactions to be considered are always reduced to two types of interactions: these are between the liquid molecules and between the liquid and the individual flavor molecules. This also explains the fact that water, as the main component dominating the proportion, specifies the boiling point of a beverage close to the boiling point of pure water. Conversely, flavorings with strongly deviating boiling behavior also boil close to the boiling point of pure water. This also makes it possible to consider the flavor substances separately from one another in terms of evaporation behavior. It also makes it clear that the volatility of the diluted flavors must never be estimated based on their boiling point!

The importance of this simplification cannot be stressed enough, as it greatly simplifies the calculation of the mixing behavior in general and in relation to evaporation processes. Going back to the example that a flavor molecule interacts like water molecules on the liquid side, it is sufficient to calculate the reduction of the flavor substance – only valid for this ideal behavior – to consider only the vapor pressures of water and flavor substance at a certain process temperature.

An influencing factor to be considered are the contents of ethanol, sugar and proteins. The literature confirms that in the present concentration ranges of up to 15 % carbohydrate concentration in wort there is no significant influence on evaporation [3, 10].

As a result, very few flavor substances behave ideally, and it is important to detect deviations from this ideality. The activity coefficient is used for this. A vapor pressure describes the behavior of a substance and is a substance property. The activity coefficient is to be understood as a correction factor for the mixture. For an ideal mix it is 1, for real mixes it deviates upwards or downwards. For the present concentration range of the flavors, this parameter has the positive property that it is mainly temperature dependent. This is clearly justified by the idea of highly diluted molecules: whether their number fluctuates upwards or downwards has no further influence due to the dilution; a flavor molecule (solute) is still only surrounded by water molecules (solvent).

These introductory words serve to arouse a feeling for the problem of mixing. In summary, the molecular concept allows to understand the mixing behavior of highly diluted flavor substances in an aqueous solution. In the following thermodynamic section, this understanding is substantiated with the necessary equations.

To classify the necessary dilution range, it is assumed that it begins with small molecules with flavor concentrations of less than 10^{-3}. For larger molecules such as hydrocarbons, a range from 10^{-4} to 10^{-5} can be assumed [9].

An evaporation process without reaching the boiling point driven by the concentration different between fluid and gas phase (similar to a wet T-shirt drying process), as it occurs when the Hard Seltzer vessel is emptied, means the transition of a component from the liquid phase to the gaseous state. If a mixture of two components is considered, the question arises whether the compositions of the phases differ. The answer to this is volatility. As described before, a dynamic equilibrium is established between the concentration of volatile substances in the vapor and liquid phase when the vessel is emptied. This phase equilibrium is stationary, at the same temperature ***T*** and the constant total pressure ***P*** in the liquid ***L*** and the vapor phase ***V*** [3].

$$T^L = T^V \quad \text{(Eq. 1)}$$

$$p^L = p^V \quad \text{(Eq. 2)}$$

The decisive variable for the concentrations in the gas phase and liquid is the partial pressure $\boldsymbol{p_i}$. The partial pressure corresponds to the part of the total pressure ***P***, characterized by the proportion of a substance $\boldsymbol{y_i}$ (equation 3). The partial pressure $\boldsymbol{p_i}$ of a component ***i*** is identical to the volume percent of a substance in an ideal gas mixture. Dalton's law is [3], [7]:

$$p_i^V = y_i \cdot P \quad \text{(Eq. 3)}$$

Equivalent to this is the law for ideal solutions with the substance fraction of the liquid ***x*** and the saturation vapor pressure p_i^* according to Raoult using equation 4.

$$p_i^L = x_i \cdot p_i^* \quad \text{(Eq. 4)}$$

Due to the described non-ideal behavior of the juices, the relationship must be extended by an activity coefficient ɣ, which records and quantifies the deviation from an ideal solution.

$$p_i^L = x_i \cdot \gamma_i \cdot p_i^* \quad \text{(Eq. 5)}$$

This activity coefficient is very complex in many applications and can only be determined with complex devices, methods and databases of thermodynamics. For the present case of infinite dilution and the non-existent interactions, the activity coefficient is simplified and therefore does not depend on the composition of the solvent, but only on the temperature of the substance mixture [3], [4]. When the thermodynamic equilibrium is established, the partial pressure in the liquid and gas phase is also identical.

$$p_i^L = p_i^V \quad \text{(Eq. 6)}$$

The phase equilibrium enables Dalton's and Raoult's laws to be equated. This is known as Raoult's expanded law for real liquid mixtures.

$$y_i \cdot P = x_i \cdot \gamma_i \cdot p_i^* \quad \text{(Eq. 7)}$$

However, the same partial pressure does not necessarily mean an identical concentration in the gas phase and in the liquid. The distribution factor K_i is therefore added as a further parameter. This describes the concentration ratio of a component in the vapor phase to the component in the liquid phase. In simple terms, a comparison is made between which substance is more volatile and which is more enriched on the gas side. It is substance-specific and, as table 1 shows, temperature-dependent. Since this value indicates whether a substance occurs mainly in the gas or in the liquid, the distribution factor is also referred to as the absolute volatility ***K***.

$$K_i = \frac{y_i}{x_i} = \gamma_i \cdot \frac{p_i^*}{p} \quad \text{(Eq. 8)}$$

Many of the flavorings found in beverages are highly volatile (K > 1). They are characterized by the fact that they pass into the gas phase in a higher concentration in terms of quantity. They therefore have a high absolute volatility. In relation to the flavored beverages, one now considers the binary mixture system of the solvent ***j*** and the respective flavoring substance ***i***. The so-called relative volatility α [3] results from the quotient of the two separation factors:

$$\alpha_{i,j} = \frac{K_i}{K_j} = \frac{y_i / x_i}{y_j / x_j} = \frac{\gamma_i \cdot p_{0i}^{LV}}{\gamma_j \cdot p_{0j}^{LV}} \quad \text{(Eq. 9)}$$

The relative volatility describes the behavior of the individual flavor substances in the mixture.

This relative volatility is to a good approximation in infinite dilutions equal to the distribution factor ***K*** and serves as a basis for calculation with regard to the depletion of the flavor substances when the vessel is emptied.

2 Material and methods

Due to the often non-existent substance-specific constants, a method has proven to be useful for determining the relative volatilities [3, 4], which consists in determining the relative volatility of a component via its change in concentration on the liquid side in relation to the amount of water that is also evaporating.

The derivation of the equation for calculating the relative volatility α is listed below.

Based on a single-stage evaporation / evaporation (Fig. 4, see page 138) of a highly diluted binary solution in a closed system, the following material balance results. Here, in the total amount of substance, ***x***, ***y*** are the proportions of the amount of substance in the liquid and gas phase.

$$nx_1 = y_1 dn + (x_1 - dx_1)(n - dn) \quad \text{(Eq. 10)}$$

Here, in the total amount of substance, ***x***, ***y*** are the proportions of the amount of substance in the liquid and gas phase. After transforming and separating the variables, the following equation results:

$$\frac{1}{n} dn = \frac{1}{(y_1 - x_1)} dx_1$$

With the assumption of infinite dilution from equation 10, the following applies:

$$y_1 = \alpha_{1,2}^{\infty} \cdot x_i \quad \text{(Eq. 11)}$$

After inserting this condition for y_1 in equation 11, one obtains the following expression for the relative volatility after integration and conversion [3, 13].

$$\alpha_{1,2}^{\infty} = \frac{\ln\left(x_{1,1} / x_{1,0}\right)}{\ln\left(n_1 / n_0\right)} + 1 \quad \text{(Eq. 12)}$$

Table 1 experimental set-up for volatility attempts

sugar [%]	ethanol [%]	Quantity suspension [L]	head-space [L]	sugar (juice) [g]	Quantity solution [L]	sugar add on [g]
0 %	5 %	9	41	810	0,5 EtOH + 1,5 H_2O	0
0 %	0 %	9	41	810	2	0
15 %	0 %	9	41	810	2	690
25 %	0 %	9	41	810	2	1690
25 %	0 %	36	14	3240	8	6760

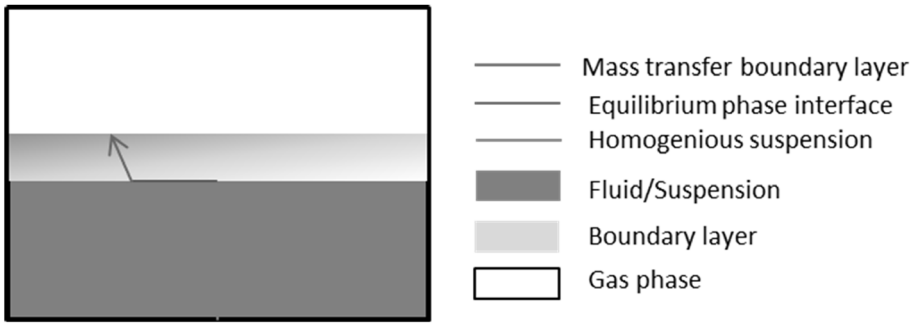

Fig. 4 Representation of the gas- and liquid-side concentration gradients

To determine the concentration differences $x_{1,1} / x_{1,0}$ of equation 12, stainless steel barrels with a nominal volume of 50 liters were filled with a flavor suspension under a protective gas atmosphere. The suspension was enriched with differing sucrose and ethanol concentrations to maintain the influence of the ingredients on the activity coefficient and thus on the volatilities.

The barrels were pressurized with nitrogen in order to minimize the oxidative influence, an initial sample was taken and sealed airtight. After the 24 hours have elapsed, it is assumed that equilibrium is fully established in terms of water vapor saturation and flavor equilibrium. The amount of water in the air is calculated according to the Mollier relationships, which are assumed to be known, and yields the concentration difference of the solvent n_1 / n_0 from equation 12. The samples were examined for the key flavoring substance limonene, which is important for the beverage industry, using the SAFE method [10].

The concentration differences enable the volatilities to be calculated using equations 12 and illustrate the relevance of the problem of a batch manufacturing process.

In addition to this until here described small-scale experiment, while an industrial batch vessel was being emptied in the outlet, depending on the increasing headspace, the flavor concentration was measured and compared with the theoretical calculations.

To clarify the importance of the activity coefficient and the incomplete state of the art, the evaporation behavior of benzaldehyde, which is important in addition to limonene, is theoretically examined in a supplementary experiment. Two vessels are heated under atmospheric conditions and open to the ambient air. Vessel A contains pure benzaldehyde and vessel B contains pure water. Just below the atmospheric boiling point of water at approx. 100 °C, both vessels are kept hot at this temperature for a certain time. The decrease in mass is determined at defined intervals.

3 Results and discussion

The result of the evaporation behavior of benzaldehyde is that more water than benzaldehyde evaporates in a certain time. The explanation is simple: the vapor pressure of water is almost 1 bar due to its proximity to the boiling point, that of benzaldehyde is only about 0.0084 bar. The mass flow of water is higher than that of benzaldehyde. A calculation of the volatility of benzaldehyde using the vapor pressures leads to a value of approx. 0.008. Benzaldehyde is clearly the high boiler. Experiment and theory clearly agree.

If, in a second experiment, the flavoring substance is infinitely diluted in water and this mixture is exposed to the same test conditions in a vessel, then, according to the previous result, a much stronger evaporation of water would be expected. Based on the determined and calculated volatility value of 0.008, a concentration of benzaldehyde would be expected. However, if this experiment is carried out, the surprising result is that more benzaldehyde than water evaporates. The volatility of benzaldehyde at approx. 100 °C is approx. 22 [3]. Benzaldehyde in infinite dilution is now the low boiler. A single estimation of the evaporation behavior of benzaldehyde based on volatility values calculated from vapor pressures leads to a significant misjudgment of the actual decrease and too often found in the literature [5] volatility values that are far too low. An activity coefficient is required that describes the deviation from the ideal considerations.

When emptying the vessel, another paradox affects the correct assessment of a reduction in flavors through the process. Relative volatilities usually increase with decreasing temperature. This is justified by the relationship between the vapor pressures of the flavor and the solvent, which increases with falling temperatures [4]. The importance of the activity coefficient should be mentioned again here, which is greatly simplified in relation to the interactions in the context of infinite dilution but is still temperature dependent. Table 1 shows this behavior for some flavors [4]. The discrepancy between the relative volatilities and the simple partial pressure difference in the last column is clear. The exact test procedure for determining the volatilities can be found in the literature [1], [3].

The results of the evaporation behavior of limonene show considerable reductions in the key flavor in figure 5.

Table 2 relative volatilities of flavors as a function of temperature [1], [3]

flavors	α 20 °C	α 90 °C	α 100 °C	$p_{flavor}/p_{solvent}$ 100 °C
2-Methylbutanal	151,3	69,5	64,7	1,3
3-Methylbutanal	108,5	65,6	62,4	1,2
Benzaldehyd	133,0	27,4	21,6	0,1
2-Furfural	55,4	9,4	7,7	0,1
DMS	4600,0	131,0	75,6	5,5

Table 3 calculated volatility values as a function of the juice matrix

sugar [%]	ethanol [%]	calculated relative volatility Limonen at 20 °C
0 %	5 %	3,8
0 %	0 %	7,9
15 %	0 %	15,1
25 %	0 %	23,6

A clear decrease in the reductions in limonene with increasing sucrose concentrations can also be seen. The addition of ethanol, on the other hand, has a volatility-reducing effect (Fig. 6).

In table 3, the volatility relative to the solvent (flavor suspension) in the various suspensions has been determined according to equation 12 and the presumed known moisture behavior of gas [12].

Compared to the volatilities listed in table 2, limonene ranks among the medium-volatile substances and, as expected, is strongly dependent on the matrix (sucrose, ethanol content).

In a further experiment, the decrease in the concentration of limonene was measured during a vessel emptying of a suspension like that in the barrel experiments with 15 % added sucrose. According to the principles from figure 2 [2], a 75 m³ vessel was filled to 10 % free head space. An agitator at the bottom of the vessel was active at moderate speed (n = 10 1/min) throughout the experiment. The emptying process lasted 60 minutes – during this, samples were taken from the vessel outlet and their flavor concentration was measured.

The measured data are compared with the basics described in chapter 1 [2] and the volatilities calculated for limonene in table 3. It is noticeable that the measured values consistently result in somewhat higher losses than the theoretically calculated ones. The deviation can easily be explained by oxidative and volatility-based losses during sampling and measurement.

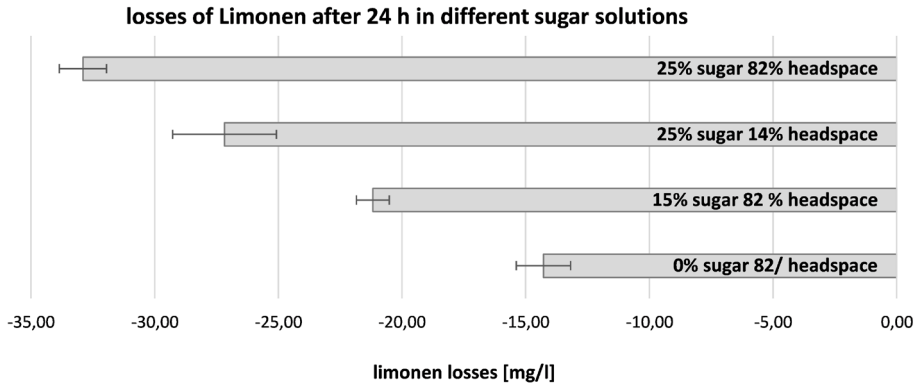

Fig. 5 Losses of limonen in different sugar solutions after 24 hours with closed storage

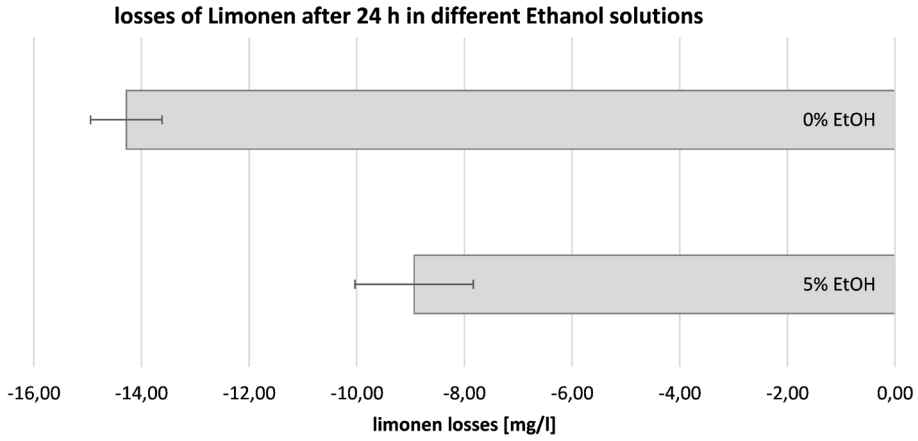

Fig. 6 Influence of ethanol on the reduction of limonene in closed storage

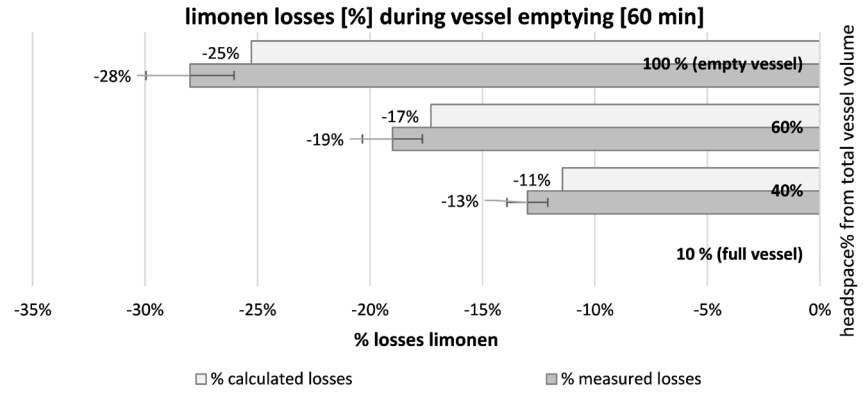

Fig. 7 Loss of flavor during vessel emptying

4 Conclusions

When producing flavor-intensive suspensions such as Hard Seltzer, mixed beer beverages, carbonated soft drinks or juices, the flavor concentration always changes due to the equilibrium setting of the concentration differences in the batch process. As a rule, the product is conveyed directly from the preparation vessels to the filler. First of all, there is a constant depletion when the beverages are stored in the headspace of the batch vessels. However, emptying the mixed suspension from the vessel affects the product quality much more, since the depletion is not constant. Accordingly, the product processed at the beginning has a higher concentration of specific flavors than the product that is filled at the end of the vessel emptying. These differences are particularly annoying to the consumer, as constant quality and standardized products are expected. The results make it clear how the product quality about the flavor concentration deteriorates as the vessel becomes empty. Viewed objectively, with a batch-supported process, the consumer receives a much more flavor-intensive drink at the start of production than at the end of production.

The volatilities of the specific flavors, considering the activity coefficient, are many times higher than previously assumed [2,5] and as the sole quotient of the vapor pressures suggests. The influence of the flavor suspension can be clearly seen. Increasing

sucrose concentrations steadily increase the volatilities in the examined suspension. This means that beverages with a high (residual) sugar content must be processed much more carefully than products with lower sucrose contents. Ethanol, with its non-polar carbon chains, has a volatility-reducing effect and helps to reduce the loss in a batch process, especially with the Hard Seltzers.

If a batch process cannot be avoided for economic and processing reasons, the following deductions from the results must be observed to minimize the losses.

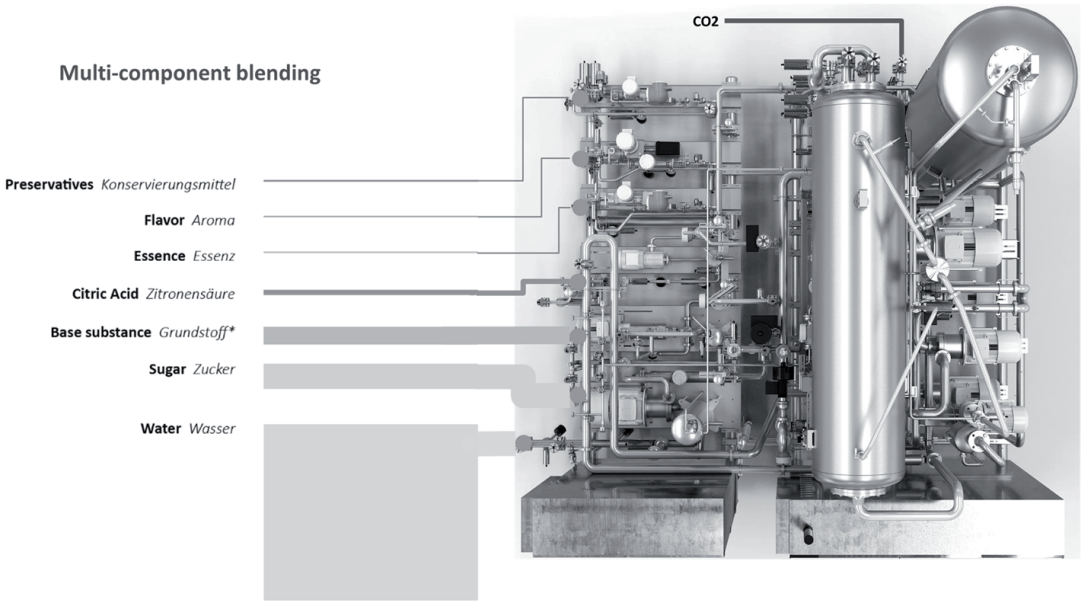

Fig. 8 Multicomponent inline blender [11]

- vessel dimensions precisely adapted to the batch sizes
- always fill the vessel as full as possible
- just in time production
- no long downtimes
- reduce the number of vessels and pumping to a minimum
- depending on the volatility behavior, process at low temperatures if possible
- volatilities increase with decreasing temperature).
- during emptying, do not stir / pump over etc. to limit the depletion to the uppermost layers of the liquid level.

The results show surprisingly clearly that the production of flavor-intensive drinks should always take place with an inline mixing system as shown in figure 8. Due to the separate and closed supply and admixture of the individual components in the carrier liquid (beer, water), this process hardly offers an opportunity to establish a concentration equilibrium and guarantees a constant flavor profile during production [11].

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