



# Tackling Foam-Based Process Disruptions in Spirit Distillation by Thermal Energy Input Adaptations

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## Abstract

Process impairing foam formation occurs regularly in batch distillation devices of the spirit industry. It negatively influences process and product quality. Up to now, such foam-related problems have not been in the focus of scientific investigations. This study aimed at preventing impairing foam formations by adapting the thermal energy input in fruit and grain mash distillations in larger scale batch distillations. The results showed that a reduction of the thermal energy input to  $43 \pm 1 \text{ W}\cdot\text{L}^{-1}$  during the initial heating of the mash leads to less flooding of the distillation apparatus and to a higher concentration of lower boiling compounds like methanol, acetaldehyde, and ethyl acetate as well as ethanol in the first fractions of the distillates. A standard process time and less energy consumption could be achieved by increasing the energy input again after prior reduction. However, this led to a reduction of the ethanol concentration in the distillate fractions of up to 4.3%vol, also most severe in the first fractions. A significant influence on analyzed volatile compounds in the distillate besides ethanol could not be detected. This is the first study that uses defined thermal energy input adaptations for foam management in larger scale distillation devices. The results lead the way to a more efficient distillation process with less foam formation.

**Keywords** Foam management · Distillation control · Spirit drink production · Process design · Energy efficiency

## Introduction

In fermentation technology, foam formations are generally considered an undesired side effect (Dorsey, 1959; Stanbury et al., 1995). Foam formation can lead to over foaming of fermentation vessels or disturbances during subsequent process steps of the fermented substrates (Murray, 2014). With respect to distillation processes, the presence of excessive foams leads to the carryover of foams onto separation trays, product contamination, and increased cleaning demands. The effects are associated with reduced separation efficiency and process disruptions (Kister, 2003; Miller, 2019; Schidrowitz & Kaye, 1906). When it comes to column malfunctions in the distillation industry, foaming plays a major role (Kister, 2014). In spirit drink production, certain raw materials are referred to be especially prone to excessive foam formation, such as

cherry, wine yeast, Bartlett pear, and grain mashes (Pieper et al., 1977). Aside from this descriptive information on foam formation capacities of different mashes, Heller and Einfalt (2021) demonstrated in laboratory experiments that the polysaccharide pentosan found in rye has a major impact on foam formation in distillations of rye mashes. Additionally, it is well-known that besides polysaccharides, proteins play a crucial role in the physical stability of food foams (Dickinson, 1998, 2003; Nunes & Coimbra, 1998; Prins, 1988; Ye, 2008). However, in spirit drink production, comprehensive researches on substrate-based factors influencing foam formations in distillations are still missing.

To reduce foam formation during distillation, several measures have been applied. For instance, manufacturers introduced adaptations to their distillation devices such as increased pot still headspace volume and foam retention installations (Pieper et al., 1977). This, however, increases manufacturing costs. Such adaptations already have a long history. For example, the elongated swan necks in Scottish pot stills in whisky distilleries were originally invented to prevent the transfer of rising foams into the product stream (Schidrowitz & Kaye, 1906). Unfortunately, larger distillation devices have a higher surface area, which is associated

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with higher process costs due to additional surface area heat radiation.

In addition to these aspects, operators of distillation plants take their own actions to prevent undesired foam formations. Chemical defoamers based on silicone or mineral oil, also called anti-foam agents, are widely used for this purpose (Miller, 2019). The negative aspect of defoamers is their disposal with the stillage without special treatment. They end up in the environment, where they can cause undesired ecological effects (Routledge et al., 2014).

Detailed information on process-related solutions for proper foam management in distillation is scarce and for the most part based on heuristic recommendations. Heller and Einfalt (2021) showed that 90% of foam formation took place at mash temperatures  $\geq 89.5$  °C. Pieper et al. (1977) recommended “reduced thermal energy input” in order to diminish excessive foam formation. They found it beneficial to “slow down the heating process in the foam-critical temperature range.” However, no additional information is given to define the thermal energy input magnitude or foam-critical temperature range. Therefore, the handling of foams is still dependent on operator experience. Extensive research is required to support new and experienced distillation plant

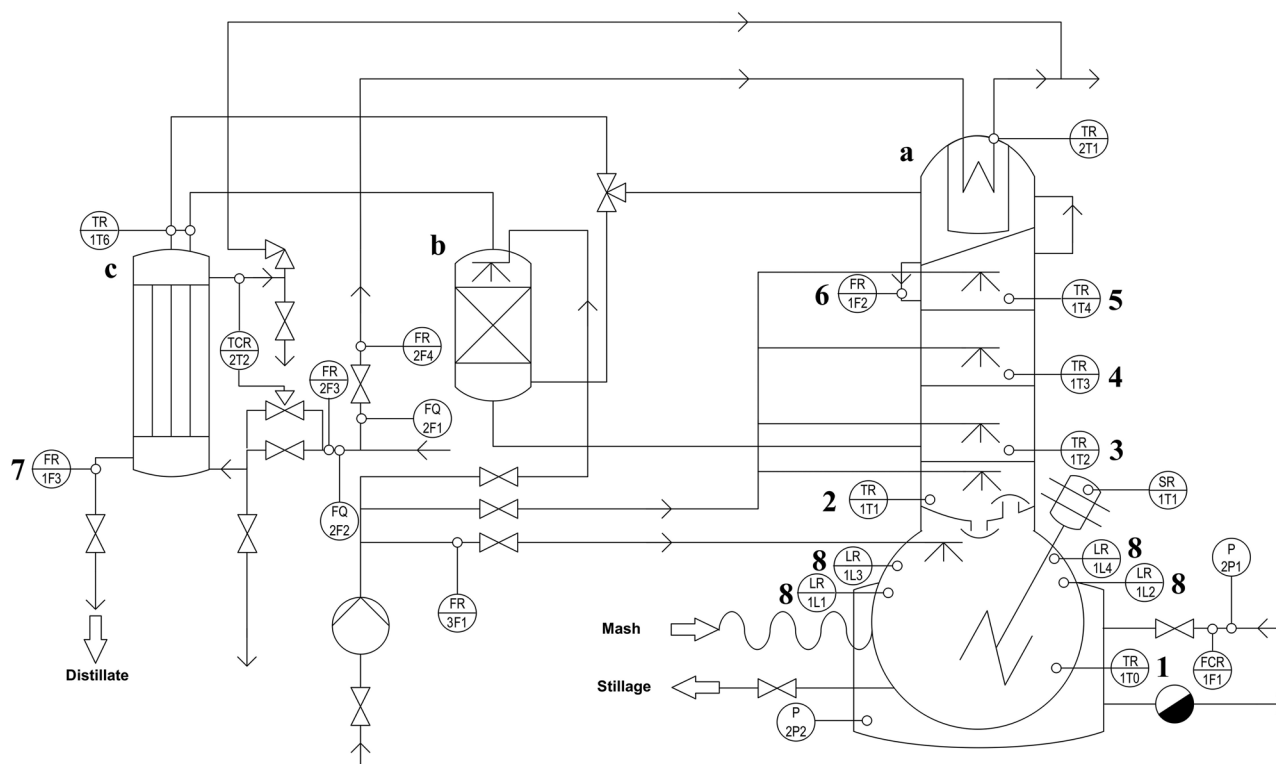
operators in handling foam formations and in assessing the impact of anti-foam measures on their distillate.

Therefore, it would be desirable to define parameters that enable a foam-resilient distillation process with low additional costs for manufacturers and operators. To close this knowledge gap, this study investigated the effects of different heating profiles on foam formation in larger scale distillation processes based on fruit and grain mashes. The study also evaluated effects on process time, energy consumption, and volatile compound composition in the resulting product.

## Materials and Methods

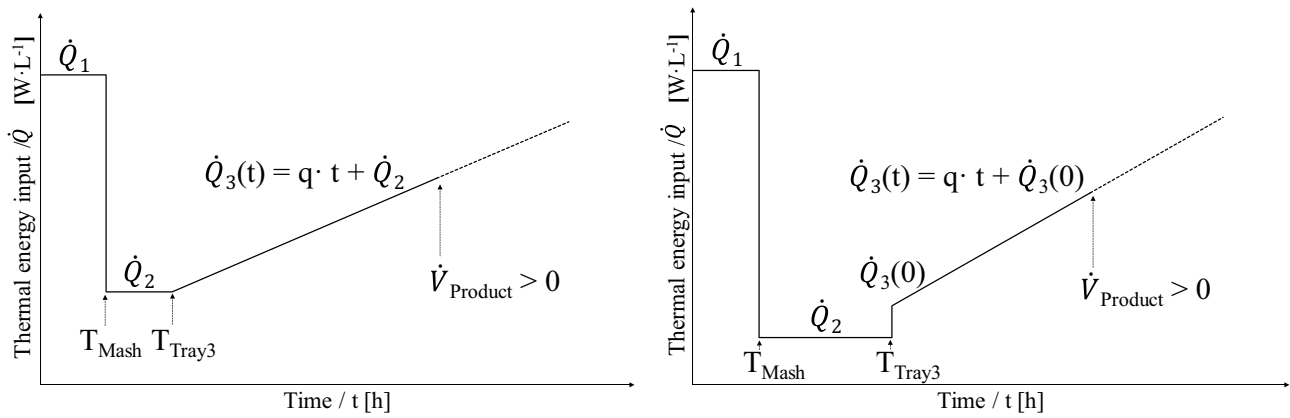
### Distillation System

A steam-heated 120-L batch copper distillation device equipped with a rectification column (Carl GmbH, Eislingen, Germany) on top was used for the distillation experiments (Fig. 1). The attached rectification column was equipped with three sieve trays, a partial condenser at the top, and a foam retention installation at the bottom. The distillation device also contained four foam-detecting



**Fig. 1** Instrumentation diagram of the batch distillation system equipped with rectification column (a), copper catalyzer (b), and product condenser (c). 1: Temperature sensor “Mash.” 2: Tempera-

ture sensor “Headspace.” 3: Temperature sensor “Tray 1.” 4: Temperature sensor “Tray 2.” 5: Temperature sensor “Tray 3.” 6: Flow sensor “Reflux.” 7: Flow sensor “Product.” 8: Level sensors “Foam height”



**Fig. 2** Heating profile of experiments in the first (left) and second (right) test series

Switzerland) installed in the reboiler at different heights positioned, 5 cm, 10 cm, 15 cm, and 20 cm above the mash surface. Excessive foam formation, which reached the foam retention installation or the trays, was logged manually. The distillation device was additionally equipped with eight temperature sensors (PT100) positioned in the reboiler, at different heights of the rectification column, in the vapor line, and in the product condenser. Further technical sensors (Coriolis Micro Motion H Series, Emerson Electric Co., Ferguson, USA) measured product volume flow and reflux volume flow of the partial condenser. Cooling water flow in the product condenser and the partial condenser were measured via SM6120 flowmeters (ifm electronic GmbH, Essen, Germany).

The distillation device was regulated and monitored via an automated process control system (DPC500, Carl GmbH, Eislingen, Germany). It enabled the regulation and control of the thermal energy input via an electro-pneumatic steam valve (SP400, Spirax-Sarco Engineering plc, Cheltenham, UK). Besides thermal energy input, the process control system also regulated the cooling water volume flows. All technical sensors were monitored, and data were recorded every 10 s via the process control system. The measured data were averaged over 60 s.

**Experimental Setup**

Two test series were performed in order to investigate the effects of modified heating profiles on foam formation (Fig. 2 and Table 1). The basis was a common heating profile (Dürr et al. 2010), which was modified at two points (adaptation points) in the test series and served as a control.

The common heating profile started off with an initial thermal energy input of 450 W·L<sup>-1</sup> (Q-dot\_1). At a defined mash temperature of 90 °C (T\_Mash = first adaption point), the thermal energy input was reduced to 134 W·L<sup>-1</sup> (Q-dot\_2). When the top/third tray of the rectification column reached a temperature of 75 °C (T\_Tray3 = second adaption point), a steady increase of thermal energy input (Q-dot\_3(t) and q, respectively) was applied. The thermal energy input was increased until the first product ran out of the distillation device. Subsequently, the distillation device automatically regulated the thermal energy input to maintain a constant product flow of 10 L·h<sup>-1</sup>. Cooling water flow of the partial condenser was kept constant at 2 L·min<sup>-1</sup>. The distillation process was terminated, as soon as the ethanol concentration of the product dropped below 25%vol.

The first test series employed reduced thermal energy input levels to inhibit foam formation. Thus, the control

**Table 1** Process parameters for experiments in the test series

Test series number	Heating profile	T <sub>Mash</sub> (°C)	T <sub>Tray3</sub> (°C)	Q-dot <sub>1</sub> (W·L <sup>-1</sup> )	Q-dot <sub>2</sub> (W·L <sup>-1</sup> )	Q-dot <sub>3</sub> (0)(W·L <sup>-1</sup> )	q(W·L <sup>-1</sup> ·h <sup>-1</sup> )
1 and 2	Control	90	75	450 ± 1	134 ± 1	134 ± 1	46 ± 2
1	HP1.1	75	75	450 ± 1	95 ± 1	-	46 ± 2
1	HP1.2	75	75	450 ± 1	59 ± 1	-	46 ± 2
1	HP1.3	75	75	450 ± 1	43 ± 1	-	90 ± 2
1	HP1.4	90	75	450 ± 1	43 ± 1	-	90 ± 2
2	HP2.1	90	75	450 ± 1	43 ± 1	59 ± 1	80 ± 2
2	HP2.2	90	75	450 ± 1	43 ± 1	59 ± 1	121 ± 2
2	HP2.3	90	75	450 ± 1	43 ± 1	59 ± 1	161 ± 2

heating profile was modified at the first adaption point. In three experiments (HP1.1–HP1.3), the adaption point  $T_{\text{Mash}}$  was set to a mash temperature of 75 °C and the thermal energy input ( $\dot{Q}_2$ ) was reduced to 95, 59, and 43  $\text{W}\cdot\text{L}^{-1}$ , respectively. In a subsequent experiment (HP1.4), a reduced thermal energy input of 43  $\text{W}\cdot\text{L}^{-1}$  ( $\dot{Q}_2$ ) at the original  $T_{\text{Mash}}$  was applied. Because of customs restrictions concerning the duration of distillation runs, the increase in thermal energy input ( $q$ ) was higher in HP1.3 and HP1.4.

The second test series aimed at counteracting process prolongations induced by the thermal energy reduction in the first test series. It was based on the most promising heating profile of the first test series and introduced changes at the second adaption point. When  $T_{\text{Tray3}}$  was reached, the thermal energy input was raised by a certain amount ( $\dot{Q}_3(0)$ ); then, the thermal energy input ( $q$ ) was increased over time as before. Distillation experiments in the second test series started with HP2.2. Subsequent distillation experiments were performed with HP2.1 or HP2.3 due to observed foam formation in HP2.2.

Distillation experiments of the first test series were performed with 100 L rye mashes in single determination. Distillation experiments of the second test series were performed with 100 L rye mashes and 100 L Bartlett pear mashes each in duplicates.

## Sampling

The produced distillates were collected in 20 fractions for subsequent analysis. The first ten fractions contained 100 mL, while fractions 11–15 contained 200 mL and fractions 16–19 contained 1 L each. The final fraction contained the rest of the produced distillate. Distillations of Bartlett pears yielded less distillate. Bartlett pear distillates were therefore fractioned until fraction 18, while fraction 19 contained the rest of the produced distillate (< 1 L).

## Raw Materials and Mash Preparation

Winter rye was purchased from Hahn-Mühle (Ostfildern, Germany) and Bartlett pears imported from South Tyrol were received from Kaiser Destillerie-Obstweinkellerei (Salach, Germany).

Bartlett pears were shredded via fruit mill (Helmut Rink GmbH, Amtzell, Germany) and transferred to a 1000-L stainless steel tank. The pH value was adjusted to pH 3.0. The mash batch was simultaneously liquefied with 10  $\text{mL}\cdot\text{hL}^{-1}$  pectin lyase (IUB 4.2.2.10, Schliessmann, Schwäbisch Hall, Germany) and fermented with 20  $\text{g}\cdot\text{hL}^{-1}$  selected yeast strains (AROMA plus, Schliessmann, Schwäbisch Hall, Germany) over a period of 3 weeks.

Winter rye was mixed with water at a ratio of 1:4 (w/w), liquefied using 1.4  $\text{mL}\cdot\text{hL}^{-1}$  of Distizym BA-TS

(Erbslöh GmbH, Geisenheim, Germany) at 90 °C (30 min), and subsequently saccharified at 60 °C (30 min) with 5.4  $\text{mL}\cdot\text{hL}^{-1}$  of Distizym AG-Alpha (Erbslöh GmbH, Geisenheim, Germany) and 2  $\text{g}\cdot\text{hL}^{-1}$  of enzyme Tegaclast (Tegaferm, Baumgarten, Austria). Subsequently, rye mash batches were cooled to  $\leq 30$  °C and pitched with 20  $\text{g}\cdot\text{hL}^{-1}$  yeasts (Kornbrand-Premium, Schliessmann, Schwäbisch Hall, Germany). The mash batches were fermented for 3 to 4 days in 1000-L stainless steel tanks at pH 3.8.

In order to quantify only the effects of the changed heating profiles on the process and the distillate composition, it was necessary to exclude influences by differing mash characteristics. Therefore, each test series was performed with the same mash batch.

## Substrate Characteristics

In order to exclude temporal changes of substrate characteristics in mash batches between distillations, all drawn mashes were sampled before distillation and analyzed for specific substrate characteristics in triplicates. Samples were stored at  $-20$  °C prior to analysis.

Substrate characteristics included quantification of ethanol concentration, performed according to Senn and Pieper (1996). Protein concentration was analyzed using the method by Bradford (1976). Total carbohydrates were quantified via phenol–sulfuric acid method (Neilson et al., 2017). pH and conductivity were measured via multimeter (HQ40D, Hach, Loveland, USA). Viscosity determination was performed after particle extraction ( $> 0.45$  mm) with a rotary rheometer (MCR 92, Anton Paar, Ostfildern, Germany) equipped with a concentric cylinder system based on method DIN 53,019 (Deutsche Institut für Normung e.V. 2009). Viscosity determination was performed at shear rates from 0.1 to 1000  $\text{s}^{-1}$  at 20 °C. In addition, dry matter (DM) and ash content of the mashes were determined according to Sluiter et al. (2008).

## Distillate Product Analysis

All distillate fractions were analyzed for ethanol concentration via standardized density determination by a u-tube oscillator (DMA 4100 M, Anton Paar GmbH, Ostfildern, Germany). Ten volatile compounds (Table 2) were analyzed using a GC-FID (GC-2010, Shimadzu, Kyōto, Japan) equipped with a headspace autosampler (HS20, Shimadzu, Kyōto, Japan) and an Rtx-Volatiles column (Restek Corp., Bellefonte, PA, USA). A 5-point calibration of all analyzed substances was performed ( $R^2 \geq 0.99$ ). All samples were water-diluted to 40%vol ethanol prior to GC analysis.

**Table 2** Analyzed volatile compounds in the fractionated distillates

Number	Substance
1	Methanol
2	Acetaldehyde
3	1-Propanol
4	Ethyl acetate
5	3-Methyl-1-butanol
6	2-Methyl-1-butanol
7	1-Butanol
8	Acetaldehyde diethyl acetal
9	2-Methyl-1-propanol
10	1-Hexanol

### Statistical Analysis

Pearson correlation was applied to ascertain significant correlations of thermal energy input with volatile compound concentrations of the distillates ( $p \leq 0.05$ ). One-way analysis of variance (ANOVA) was performed to determine significant changes in substrate characteristics of mash batches between distillation experiments. All statistical analyses were performed using SPSS Statistics (V.25, IBM, Chicago, USA).

## Results and Discussion

### Substrate Characteristics

Analysis of mash samples taken prior to distillation showed no significant differences between mashes within a mash batch. This shows that the mashes were not significantly altered during storage between the distillation experiments. The determined substrate characteristics showed deviations of up to 6.25% (Table 3, viscosity curves not shown). We attribute these deviations to the non-homogeneous nature of the substrate, as well as to the limits of accuracy of the

measurements. No alteration in ethanol content and viscosity indicated a completed liquefaction and fermentation of the mash batches prior to the distillations. Therefore, it can be assumed that differences in distillation occurred exclusively due to changes in the process conditions. The obtained results of the substrate measurements were in accordance with data from the literature (Hansen et al., 2004; Pieper et al., 1977).

### Effects of Different Heating Profiles (First Test Series)

#### Process and Foaming

Distillation with the control heating profile resulted in excessive foam formation > 20 cm and led to over foaming onto the first tray of the column (Fig. 3a). Distillations performed with heating profiles HP1.1 and HP1.2 did not result in reduced foam formation (data not shown). Both experiments showed foam formation > 20 cm. Furthermore, both heating profiles also led to excessive foam formation, which reached the first tray. In addition, both heating profiles resulted in increased process time compared to the control of 39% and 70%, respectively and in increased energy consumption of 6.8–10.9%. In HP1.3, a reduction of the thermal energy input ( $\dot{Q}_2$ ) to  $43 \pm 1 \text{ W}\cdot\text{L}^{-1}$  was applied. This resulted in reduced foam formation (Fig. 3b), where the foam height fluctuated within 10 to 20 cm. However, this heating profile showed an 86% extension of the process time and resulted in an increased energy consumption of 14.6%.

To counteract the extension of the process time, the first adaption point  $T_{\text{Mash}}$  was shifted from 75 °C mash temperature to 90 °C in heating profile HP1.4 (Fig. 4). This reduced the process time extension from 86% (HP1.3) to 63%, while maintaining a similar foaming behavior as in heating profile HP1.3. However, HP1.4 showed a higher energy consumption of 19.5% compared to the control. Excessive foam formation and over foaming onto the trays did not occur.

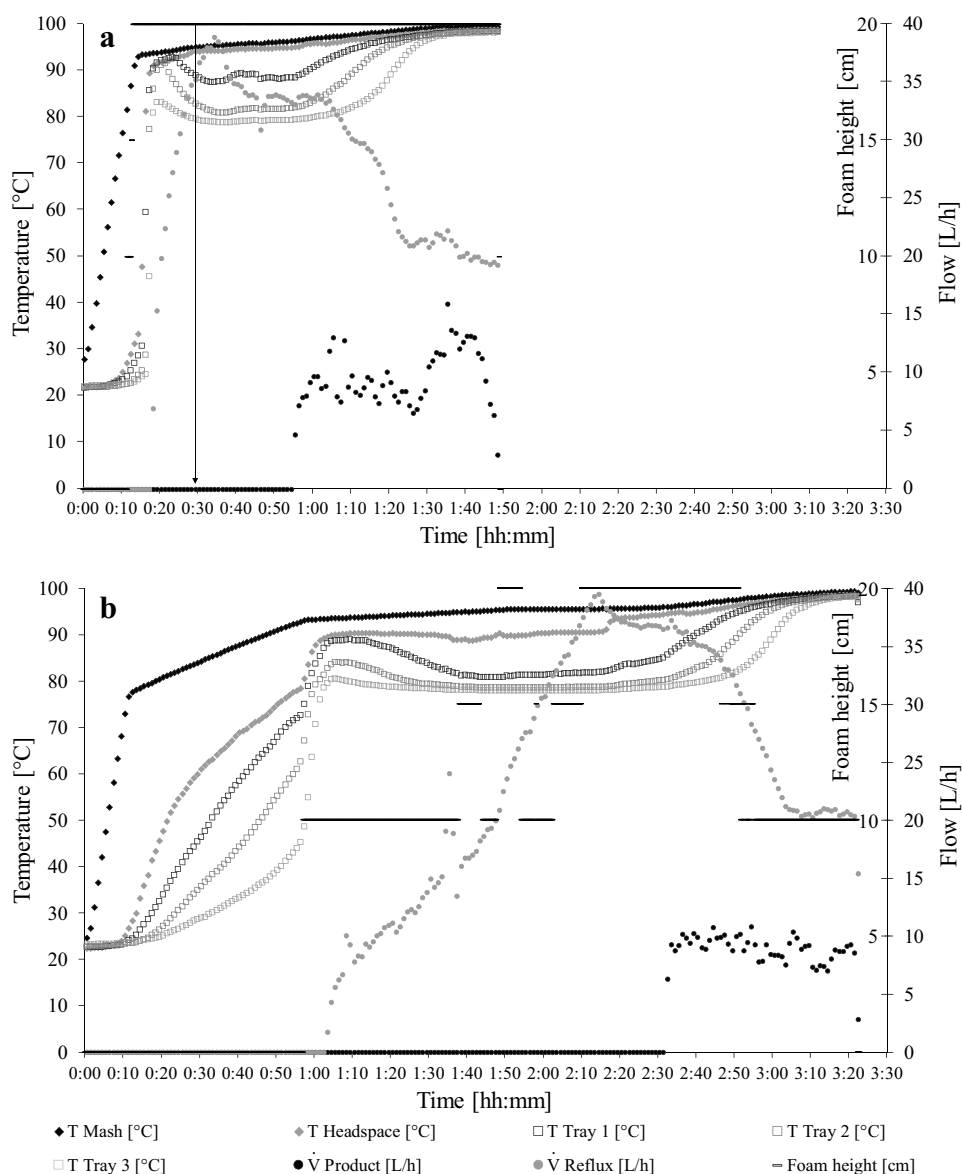
**Table 3** Mean values and standard deviations of substrate characteristics of each mash batch

Properties	Unit	Rye (batch 1) ( $n = 5 \times 3$ )	Rye (batch 2) ( $n = 6 \times 3$ )	Bartlett pear ( $n = 6 \times 3$ )
pH	-	$3.7 \pm 0.1^a$	$3.5 \pm 0.1^b$	$3.3 \pm 0.0^c$
Conductivity	$\text{mS}\cdot\text{cm}^{-1}$	$2.0 \pm 0.0^a$	$2.3 \pm 0.1^b$	$2.5 \pm 0.1^c$
Ethanol concentration	%vol	$6.1 \pm 0.2^a$	$6.5 \pm 0.2^a$	$4.2 \pm 0.2^b$
Total protein	$\text{g}\cdot\text{L}^{-1}$	$0.44 \pm 0.02^a$	$0.54 \pm 0.01^b$	$0.6 \pm 0.02^c$
Total carbohydrates	$\text{g}\cdot\text{L}^{-1}$	$18.1 \pm 0.8^a$	$19.3 \pm 0.2^b$	$16.4 \pm 0.6^c$
DM	%FM	$6.7 \pm 0.2^a$	$7.1 \pm 0.1^b$	$5.6 \pm 0.2^c$
Ash	%DM	$4.8 \pm 0.3$	$4.8 \pm 0.2$	$4.5 \pm 0.1$

Different lowercase letters within the same row indicate significant differences ( $p < 0.05$ )

FM, fresh matter

**Fig. 3** Distillation performance of control (a) and heating profile HP1.3 (b). Vertical, black arrow indicates excessive foam formation to reach the first trays



The experiments of the first test series demonstrated that foam formation can be controlled by reduced thermal energy input during the initial boiling of the mash in larger scale distillation devices. This result confirms the previously mentioned heuristic recommendation of Pieper et al. (1977) and the results obtained in laboratory-scale experiments of Heller and Einfalt (2021). The heating profile HP1.4 showed the best results regarding foam formations. However, HP1.4 had the drawback of an increased process time (63%) and higher energy consumption (19.5%) compared to a common heating profile. Nevertheless, HP1.4 should be applied, if mashes with high foam formation potential are to be distilled. Otherwise, the usage of a common heating profile may result in over foaming of the mashes leading to a process stop, a time-demanding cleaning of the distillation system, and a restart of the process.

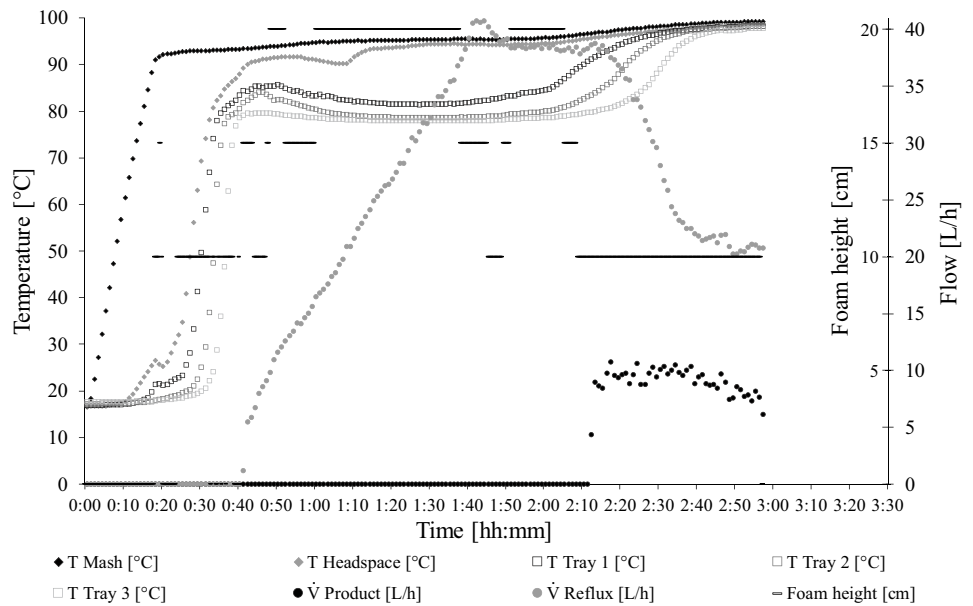
This could cause an even longer process time and a higher energy demand compared to HP1.4.

### Distillate Composition

A reduction of the thermal energy input ( $\dot{Q}_2$ ) was negatively correlated with the ethanol, methanol, acetaldehyde, and ethyl acetate concentrations in the first fractions of the distillates, i.e., the lower the thermal energy input, the higher the concentrations of the compounds in the first fractions (Fig. 5). Significant correlations of thermal energy input and ethanol (fractions 1–13), methanol (fractions 1–14), acetaldehyde (fractions 1–5), and ethyl acetate (fractions 1–4) were found ( $r = -1.000$ – $-0.951$ ;  $p = 0.000$ – $0.049$ ), respectively. The concentrations of methanol, acetaldehyde, and ethyl



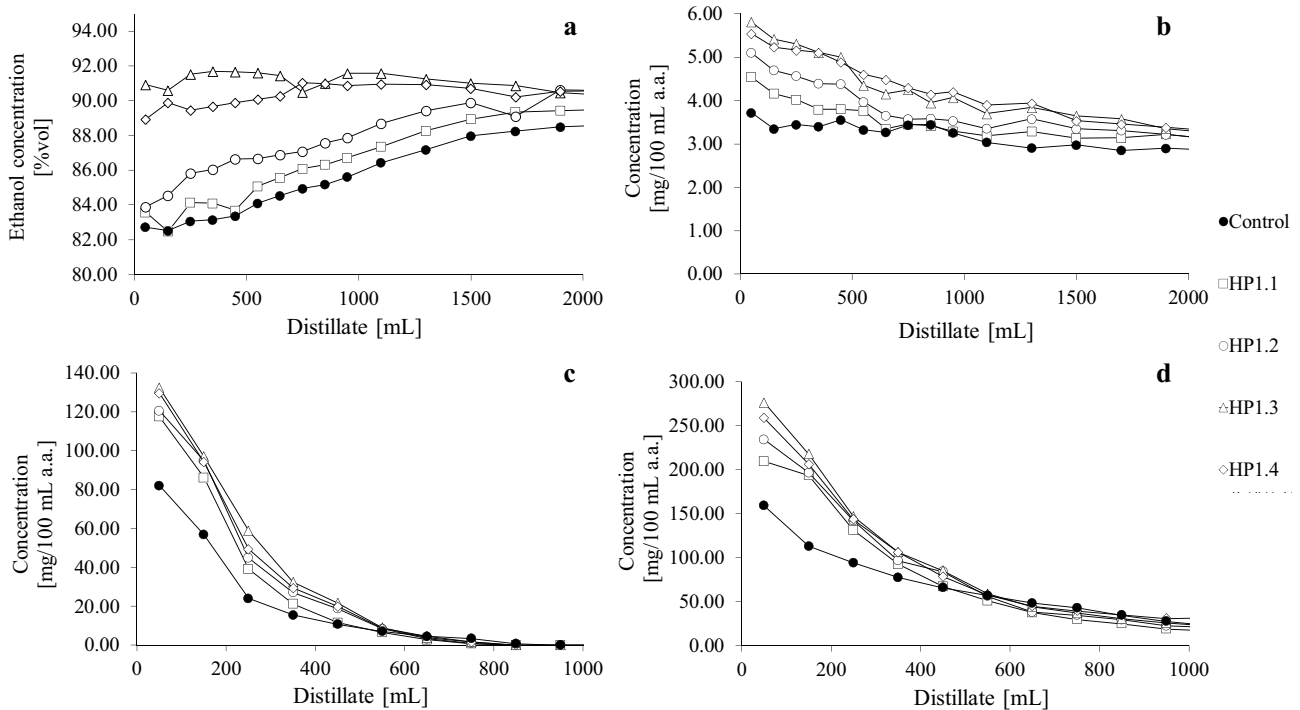
**Fig. 4** Distillation process with reduced thermal energy input at 90 °C mash temperature, HP1.4



acetate in the first fractions were 36.0%, 38.1%, and 42.4% higher in HP1.3 compared to the control. The shift of the adaption point from 75 to 90 °C mash temperature (HP1.3 and HP1.4) resulted in a maximal difference of 11.3% for ethanol, methanol, acetaldehyde, and ethyl acetate. The concentrations of all analyzed volatile compounds and their distilling behavior were in accordance with previous

reports (Aylott & MacKenzie, 2010; Douady et al., 2019; Nascimento et al., 2008; Rodríguez-Bencomo et al., 2016).

The reason for the increase of ethanol, methanol, acetaldehyde, and ethyl acetate concentrations in the first fractions of the distillates in the distillation experiments with a reduced thermal energy input appears to be a higher internal reflux in the column. High internal reflux before



**Fig. 5** Concentration of ethanol (a), methanol (b), acetaldehyde (c), and ethyl acetate (d) in the first fractions with control and modified heating profiles with reduced thermal energy input (HP1.1, HP1.2, HP1.3, HP1.4)

and around the time when the first distillate fractions are collected allows for a higher enrichment of lower boiling compounds like methanol, acetaldehyde, and ethyl acetate in the first fractions (Luna et al., 2018; Matias-Guiu et al., 2016; Scherübel, 2018; Spaho, 2017). As shown in Fig. 3, a decrease in thermal energy input increased the total volume of the reflux before the first distillate fraction was collected. The total reflux in the distillation experiment with HP1.3 was 33.8 L. The control distillation experiment showed a total reflux of 19.9 L. HP1.4 showed a total internal column reflux similar to HP1.3 (37.8 L) (Fig. 4). Because the internal reflux converges over time to a similar level of 20 L·h<sup>-1</sup> in the different experiments, the volatile compounds no longer differed in their concentration after fraction 14.

In addition to the internal reflux, other factors, like the carryover of mash onto the first tray by excessive foaming in HP1.1 and HP1.2, could in part explain the observed differences. In essence, a separation step is lost due to the carryover of liquid onto the first tray, which results in reduced rectification performance of the column (Spaho, 2017).

All in all, the experiments show that a decrease of the thermal energy input in the heating phase of the mash affects only the composition of the first distillate fractions; the later fractions seem to be largely unaffected. Because the head fractions are normally discarded and not consumed, there is no drawback in using one of the modified heating profiles with respect to the final distillate composition. No significant changes in the concentrations of the other analyzed substances could be detected in the final distillate (not shown).

## Effects of Different Heating Profiles (Second Test Series)

### Process and Foaming

In the second test series, we considered an increase in thermal energy input, when  $T_{\text{Tray3}}$  was reached, to counteract process prolongations induced by prior thermal energy reduction.

Rye mash distillations with the control heating profile resulted in foam formations > 20 cm (Fig. 6a). In both distillations, the excessive foam formations reached the first tray. In contrast to the first test series, the excessive foam formations now even reached the second tray. In the two duplicate experiments, the time when over foaming occurred differed by 1.5 min. Because of this temporal shift, only one profile is shown.

Distillations with HP2.2 (data not shown) reduced foam formation and showed no overflow onto the trays. Yet, HP2.2 showed foam formations > 20 cm and reached the foam retention device. To further reduce foam formation, subsequent distillations were performed with HP2.1. In distillations with HP2.1, foam formation was limited to the still

pot and did not reach the foam retention device. Initially, the foam height varied between 15 and 20 cm (Fig. 6b). Both heating profiles (HP2.1 and HP2.2) showed a significantly reduced extension of the process time and energy consumption compared to HP1.4. HP2.1 extended the process duration by 5.9% ± 0.5% and reduced the energy consumption by 1.9% ± 0.2% compared to the control. HP2.2 showed no increase in the process duration (< 1%) and reduced the energy consumption by 5.7% ± 0.1%.

Thus, the experiments show that the delay in process time introduced by a prolonged heating phase can be compensated by a higher thermal energy input in the later phase leading to a similar process time as in the control experiment.

The same experiments were performed with a different feedstock. Bartlett pear distillations with the control heating profile showed a high initial foam formation of > 20 cm, which was limited to the still pot (Fig. 7a). The initial foam collapsed and leveled off at 10 cm foam height. Distillations with the heating profile HP2.2 showed no foam formation throughout the process (data not shown). Distillation with HP2.3 showed foam formation of < 20 cm only in one of the duplicates (Fig. 7b). The two heating profiles resulted in an extension of the process time of 23% ± 1% (HP2.2) and 10% ± 1% (HP2.3). However, HP2.2 and HP2.3 reduced the energy consumption by 5.5% ± 0.1% and 12.1% ± 0.1%, respectively.

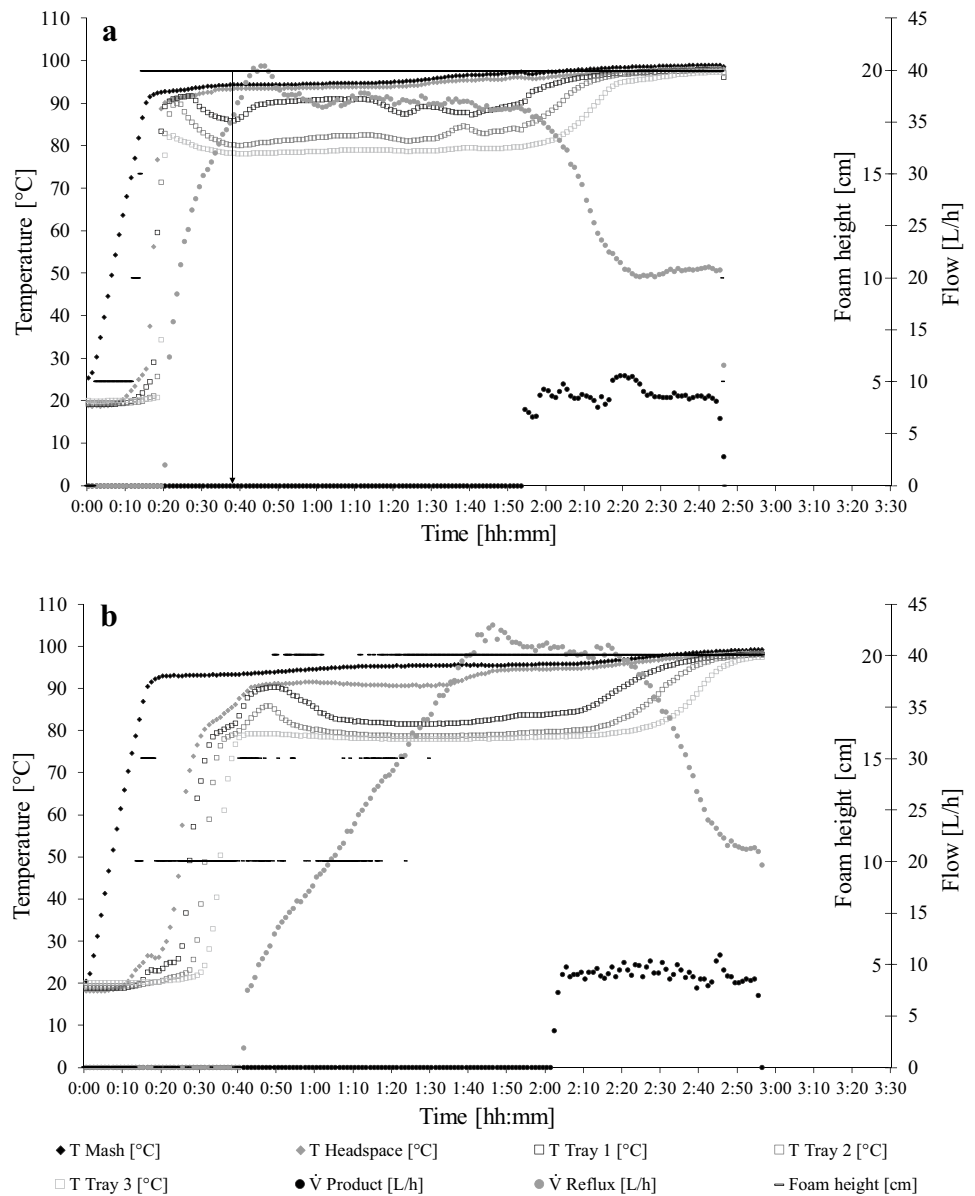
The results show that different mash types require specific heating profiles for inhibiting or reducing foam formation. Also, it became apparent that foam reduction is possible without excessive extension of process time as shown with HP2.3 in Bartlett pear and HP2.2 in rye mash distillations. Additionally, the altered heating profiles had the benefit of lower energy consumption compared to the common heating profile. We therefore concluded that the application of the altered heating profiles has no major drawbacks in terms of process efficiency, but also limits the risk of over foaming compared to a common heating profile.

### Distillate Composition

Statistical analysis of the data revealed a significant negative correlation of the ethanol concentration with a higher thermal energy input after the second adaption point in rye mash as well as in Bartlett pear distillation (Fig. 8). In distillations performed with rye mashes, significant correlations were found up to fraction 6 ( $r = -0.940$ – $-0.848$ ;  $p = 0.005$ – $0.033$ ). In distillations with Bartlett pear mash, a higher thermal energy input after the second adaption point was significantly negatively correlated with the ethanol concentration up to fraction 16 ( $r = -0.967$ – $-0.844$ ;  $p = 0.002$ – $0.035$ ). The highest ethanol concentration reduction of 4.3% vol was found in the second distillate fraction of Bartlett pear distillation with HP2.3. The reduction



**Fig. 6** Distillation process of rye mash with control (a) and HP2.1 (b). Vertical, black arrow indicates overflow of foam onto trays



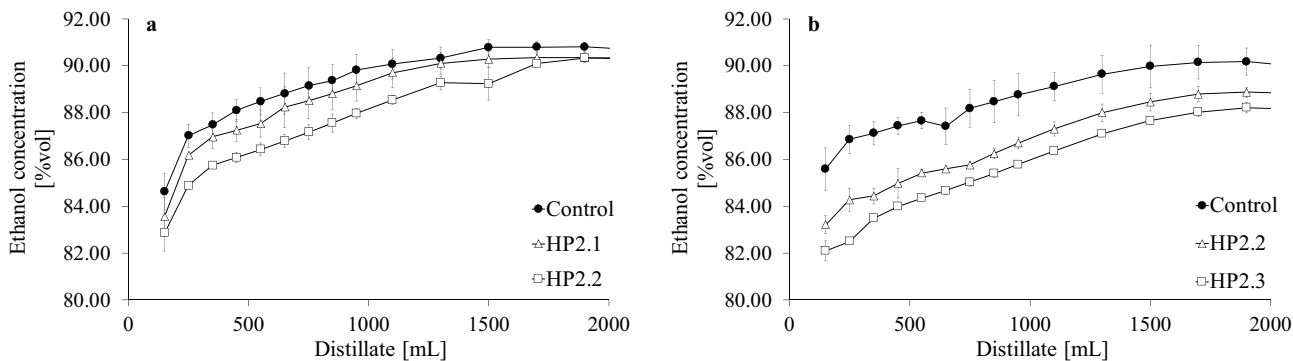
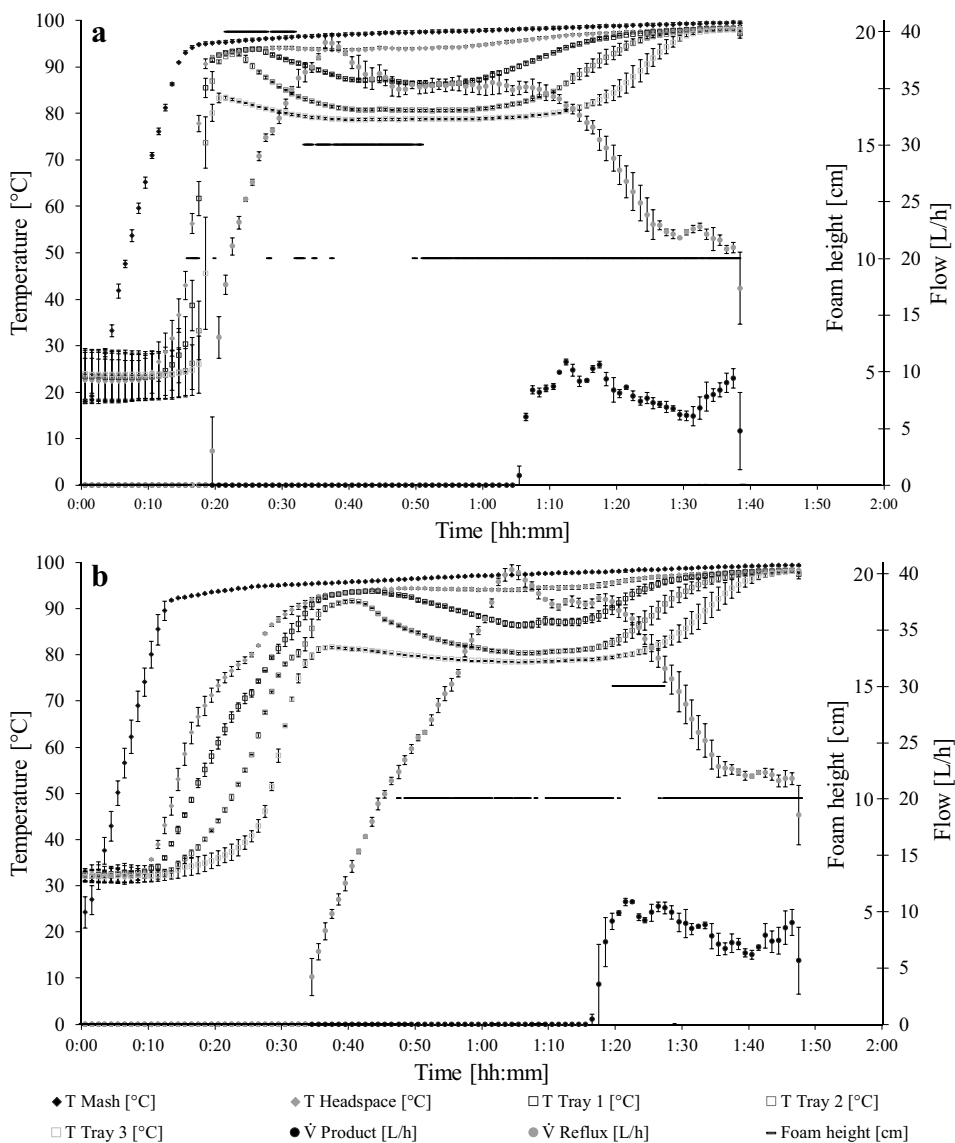
of ethanol concentration in the drinkable fractions was 1.5–3.15%vol. No significant correlations with other analyzed volatile compounds' concentrations were found.

Previously, we attributed differences of the ethanol concentration by different heating profiles to the internal column reflux and excessive foam formation, which reached onto trays. In distillation experiments with rye mashes, the control had the highest total reflux of  $55.3 \pm 2.3$  L before the first fractions. HP2.2 had the lowest total reflux of  $36.0 \pm 1.3$  L. We assume that the ethanol concentration in the control distillation would have been even higher, if not for foam formations, which reached onto the trays. This probably counteracted the effects of the higher internal reflux. In distillation with Bartlett

pear, the control showed a total reflux  $25.0 \pm 1.2$  L before the first fraction. At the same time, HP2.3 showed a total internal column reflux of  $20.8 \pm 0.9$  L. Therefore, these results were also consistent with the results of the previous experiments.

In terms of distillate composition and ethanol recovery, the altered heating profiles have the drawback of a reduced ethanol recovery compared to a common heating profile. However, the reduction in ethanol concentration was most severe in the first fraction of the distillates, which are normally discarded as heads. Considering the previously stated benefits of the altered heating profiles in terms of process and foaming, the slight reduction in ethanol recovery is economically bearable.

**Fig. 7** Distillation process of Bartlett pear mash with control (a) and HP2.3 (b)



**Fig. 8** Ethanol recovery of rye mash (a) and Bartlett pear mash (b) distillation with heating profiles control, HP2.2 and HP2.1 or HP2.3

## Conclusion

Here, we demonstrate that foaming of fruit or grain mashes during distillation can be reduced or even prevented by reducing the thermal energy input in the initial heating phase of the mash. The altered heating profiles affected only the composition of the first fractions of the distillate. Since these fractions are discarded as a head cut, significant effects on the drinkable middle run are not expected. A major drawback of our regimen was the extension of the process time. But we could show that this can be compensated by a higher thermal energy input in the later phase of distillation leading to process times similar to the control experiments. Additionally, by using altered heating profiles, the energy consumption of the distillation process could be reduced by up to 12.1%. The changes in the concentration of ethanol and the volatile compounds could be attributed to differences in the internal column reflux. Thus, a dynamic control of the partial condenser's cooling capacity should be considered to manipulate the ethanol and by-product concentrations in a desired way.

If problems with foaming occur in mashes during distillation, we recommend reducing the thermal energy input to  $43 \pm 1 \text{ W}\cdot\text{L}^{-1}$ , when the mash temperature reaches  $90 \text{ }^\circ\text{C}$ . After initial boiling, the thermal energy input can be raised to  $59 \pm 1 \text{ W}\cdot\text{L}^{-1}$  with a steady increase over time with  $80 \pm 2$  to  $161 \pm 2 \text{ W}\cdot\text{L}^{-1}\cdot\text{h}^{-1}$  depending on the used feedstock.

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**Data Availability** The data that support the findings of this study are available from the corresponding author, Daniel Heller, upon reasonable request.

## Declarations

**Conflict of Interest** The authors declare no competing interests.

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