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Optical method for porosity determination to prove the stamp effect in filter cakes

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ABSTRACT

During lautering, a filtration process in beer production, an inhomogeneous filter cake composed of different horizontal layers is formed. Fine particles settle slower than coarse particles and form a layer on top of the cake. Due to its low permeability, the fine layer acts as a stamp on the compressible bottom layer, thus resulting in cake compression and reduced flow rate (stamp effect). A method to prove the stamp effect and investigate its impact on the filtration was developed. The structure of the cake was preserved by freezing, which enabled sampling from different layers at different filtration times. An optical porosity determination (surface roughness) was established to study the compression. A predominant impact of the stamp effect compared with a less pronounced skin effect was shown. To avoid the stamp effect during filtration, a filtration technique was developed, which includes the removal of fines from the filter cake.

1. Introduction

Cake filtration is a type of solid–liquid separation process that is applied in numerous industrial sectors. It is performed after extracting valuable substances from the raw material when the liquid of interest is separated from the insoluble particles of the suspension. Among the processes of cake filtration, the lauter tun (LT) operation in beer production is extraordinary. The batch-wise process is a unique type of filtration based on the suspension to be filtered. The suspension (mash) is produced in the preceding mash tun (MT) operation by mixing the milled raw material (malt grist) with water. During the malt milling process, the husks must be preserved to maintain a porous filter cake structure in the LT. As a result, the suspension has a wide particle size distribution (Tippmann et al., 2011). The differences in the diameter (d) and density (ρ_s) of the particles lead to an irregular sedimentation behaviour when transferring the suspension from the MT into the LT, which affects the sedimentation velocity (w_f) according to

$$w_f = \frac{\rho_s - \rho_f}{18 \cdot \eta} \cdot g \cdot d^2. \tag{1}$$

The sedimentation velocity depends on the fluid density (ρ_f), gravitational field strength (g) and viscosity (η). Accordingly, coarse particles settle earlier and form a bottom layer, whereas fine particles (<500 μm) sediment later on top of the bottom layer (Engstle et al., 2017). This results in the formation of an inhomogeneous, multi-layered filter cake structure. The resulting horizontal layers differ not only in their chemical composition but also in their filtration characteristics (Hennemann et al., 2019).

Especially the fine layer determines the flow rate based on its low permeability, which results in the formation of a blocking layer on the top of the cake (Barrett et al., 1973; Bühler et al., 1996; Engstle et al., 2015). The predominant impact of the fine layer on the flow rate compared with the bottom layer distinguishes the LT operation from other filtration types. It is suggested that a skin layer next to the filter medium that regulates the filtration—as observed in other types of cake filtration (Alles and Anlauf, 2003; La Heij et al., 1996; Tiller and Green, 1973)—plays only a minor role in the LT.

In addition to its low permeability, the fine layer affects the cake filtration in another way. Based on its location on top of the cake, the drag force transmitted to the fine layer during filtration compresses the underlying compressible bottom layer similar to a stamp (stamp effect). This compression reduces the porosity of the bottom layer and results in a decrease in the flow rate. Although compression by the fines was previously suggested (Bühler et al., 1996), the stamp effect is currently not proven. Therefore, this paper aimed to investigate the stamp effect-dependent decrease in the cake porosity and the resulting flow rate reduction.

The two filtration characteristics to describe the stamp effect—flow rate (Q) and porosity—are correlated according to

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Abbreviations

LT	Lauter tun
MT	Mash tun
SF	Standard filtration

$$Q = \frac{k \cdot A \cdot \Delta p}{h \cdot \eta}, \tag{2}$$

which includes the cross-sectional area (A), pressure difference (Δp) and cake height (h). The permeability (k) is related to the porosity (ε) according to

$$k = \frac{d^2}{180} \cdot \frac{\varepsilon^3}{(1-\varepsilon)^2}. \tag{3}$$

This relation indicates that a high permeability and, thus, a high flow rate depend on a high cake porosity. The porosity is defined by the relation between the cavity volume (V_{cav}) and the total volume (V_{tot}) according to

$$\varepsilon = \frac{V_{cav}}{V_{tot}}. \tag{4}$$

For the determination of porosity in porous media, various methods are available in the literature (Bühler et al., 1996; La Heij et al., 1996; Martin et al., 2013; Mathmann et al., 2014). Although these methods can be employed to determine the filter cake porosity, there are disadvantages for an investigation of the stamp effect. For example, gravimetric porosity measurements are time-consuming and, therefore, are often only used as a reference for the calibration of another method (La Heij et al., 1996; Martin et al., 2013; Sakai and Nakamura, 2005). Another method for porosity determination based on the cake height was already applied for lautering (Bühler et al., 1996; Engstle et al., 2015), but only an average porosity of the inhomogeneous filter cake could be determined. Furthermore, the method enables porosity determination only of the peripheral filter cake area without insights into the centre of the cake. Micro-computed tomography was also employed to study the filter cake in the LT (Mathmann et al., 2014). An advantage of tomography is that insights into the centre structure of the cake and the different layers are possible. Because the sample was measured at the end of the filtration process from a dry filter cake, no insights into the cake compression during filtration were obtained.

The summary of the literature revealed that the available methods do not satisfy the requirements for investigating the stamp effect. Furthermore, the stamp effect is a unique and rather unusual mechanism among the filtration processes. Moreover, it has been neglected in the literature because it occurs exclusively during lautering in the brewing process. Therefore, the main aim of the paper was to prove the stamp effect using a new developed method for porosity determination.

The first step was the detection of the stamp effect in lab-scale filtration experiments. Using a small-scale filter, the blocking of the cake was triggered to examine the effects on flow rate and pressure drop in the filtrate pipe. The cake height measurement revealed the compression during filtration. Because the small-scale filter allowed no detailed insights into the cake compression, measurement of the porosity of the individual layers was required. Therefore, a second larger lab filter was developed to obtain filter cakes with practical dimensions (30–40 cm). The cake could be frozen to enable fixation of its structure at different filtration times. Sampling from different horizontal layers enabled the consideration of the structural inhomogeneity. A new optical method was developed to rapidly assess the porosity. The method is based on the measurement of the layer surface roughness—the arithmetical mean height of a surface profile—that correlates with the internal cake porosity. This correlation has already been validated, e. g., for the measurement of asteroid surface analogues (Sakai and Nakamura, 2005) or rocks (Rebollo et al., 1996). After the optical method was calibrated with a reference porosity measurement, it was employed to study the change in the porosity during the compression.

To prove the stamp effect, the filtration experiments were modified. Using different types of malt, the influence of the raw materials on the effect was investigated. The raw materials were selected based on their variations in the filtration behaviour. In another modification, an oxidised form of the fines was used, which decreased their permeability. The resulting accentuation of the stamp effect was investigated. The fines were removed prior to the filtration in another modification to show their requirement for the cake compression. These modifications enabled the stamp effect to be proven and revealed its predominant impact compared with a less pronounced skin effect.

Because several factors (e. g. raw material) are provided in the process, the stamp effect cannot be avoided in the conventional LT operation. Therefore, a new filtration technique was established to avoid the stamp effect by the removal of the fines. After a successful implementation in the lab scale, the technique was verified in an upscale, and the effects on the process time, yield and product quality were examined.

2. Materials and methods

2.1. Lab-scale filtration

As the first step to detect the stamp effect-dependent cake blocking, lab-scale filtration experiments were conducted using a small-scale filter (LT-1, height: 29 cm, Fig. 1a). Contrary to the practical process, the filter was used to deliberately cause the cake blocking by applying a consistent high flow rate (117 g/min) using an iPump1Q peristaltic pump (Baoding Signal Fluid Technology Co., Ltd., Baoding, China). This allowed the determination of the filtrate mass per time using a scale. The pressure in the filtrate pipe was recorded using a DTRT-AL-10 V/20 mA relative pressure transmitter (B + B Thermo-Technik GmbH, Donaueschingen, Germany) to detect the pressure drop after the cake blocking. Cake compression was examined by measuring the height (average of one central and four peripheral measurements) from the top using a folding rule.

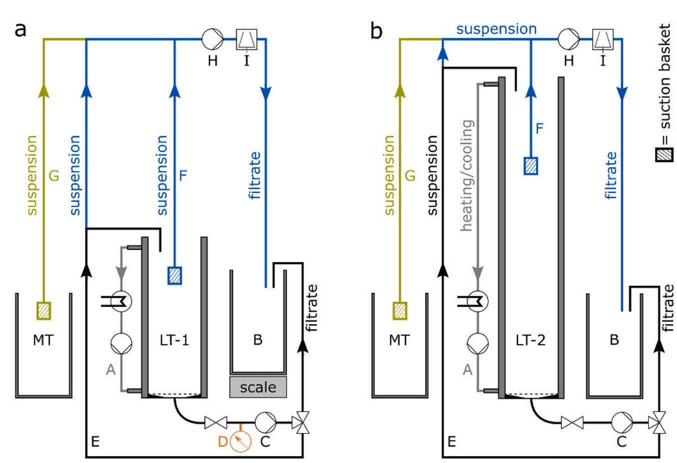

Fig. 1. Lab-scale filters: **a)** Lauter tun filter 1 (LT-1) with heating circuit (A), filtrate collector (B) placed on a scale, filtrate pipe with peristaltic pump (C), pressure sensor (D) and recirculation pipe for cloudy filtrate (E). **b)** Lauter tun filter 2 (LT-2) with an additional cooling circuit. Suspension is produced in a laboratory mash tun (MT). Suctions from LT-1/LT-2 (F, blue, LT-trial) and combined suction from MT and LT-1/LT-2 (G, green/blue, MT/LT-trial) using a suction basket connected to a peristaltic pump (H) and centrifuge (I). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

After the stamp effect detection, a second larger-scale filter (LT-2, height: 58 cm, Fig. 1b) was constructed to obtain filter cakes with practical dimensions. The cakes were used for the subsequent porosity determination to enable detailed insights into the compression. Compared with filtration in LT-1, a lower and practical filtration flow rate (43 g/min) was applied. This allowed the evaluation of the compression at different filtration states. Filtration was stopped after 56 min as a standardized process before the cake went dry, unless stated otherwise.

Both filters were heated to a practical filtration temperature of 78 °C. The filter medium (diameter: 10 cm) was overlaid with 100 mL of water to exclude the entrapped air. Contrary to the practical lautering process, no sparging water was used for cake washing, and no raking was performed to preserve the cake structure.

The setups without modifications as described in this section are referred to as standard filtration (SF) in the following.

The suspension was produced in a laboratory MT (Dinkelberg Analytics, Gablingen, Germany). Raw material (malt) was milled to grist using a DLFU disc mill (Bühler AG, Uzwil, Switzerland) at a grinding gap of 0.65 mm. Grist (LT-1: 500 g, LT-2: 1035 g) and distilled water of 60 °C were mixed at a ratio of 1:3.5. For extraction and enzymatic conversion, the suspension was heated under stirring to 62 °C (30-min rest) and 72 °C (30-min rest). After heating to a practical filtration temperature of 78 °C, the suspension was transferred to the filter.

Pre-trials revealed that oxidation exhibited a negative influence on the flow rate. Therefore, reducing agent (0.6 g/L potassium metabisulfite, Merck KGaA, Darmstadt, Germany) was added to the suspension. However, for the experiments in LT-1, no reducing agent was added to emphasise the cake blocking by the negative effects of oxygen.

After transfer to the filter, a 10-min rest allowed sedimentation of the particles. Afterwards, cloudy filtrate was recirculated back on the top of the filter in a standardised procedure until filtrate was clear (LT-1: 28 g/min for 5 min, LT-2: 21 g/min for 7.5 min).

2.2. Filter cake fixation and optical porosity determination

To obtain detailed insights into the cake compression, a new method for porosity determination was established and applied on the samples produced using LT-2. To fix the cake structure at the end of the experiment, the filter was pre-cooled to room temperature, followed by freezing overnight in a fridge.

It is known that slow freezing can affect the ultrastructure of biological particles based on the formation of ice crystals. However, no crystals were observed on a macroscopic scale during the experiments. Therefore, the obtained porosity values can be used for a comparison within the experiments in this paper.

The frozen cake was removed from the filter and cut into five horizontal layers (fine layer and four bottom layers 1–4) of equal height using a mitre-box saw. From each layer, two sample slices were cut out. The peripheral area was removed to avoid wall effects. After unfreezing at room temperature for 24 h, the porosity of the cake slices was determined by measuring their surface roughness. To correlate the values of the surface roughness with porosity, the optical method was first calibrated using a gravimetric method as reference.

The first step of the calibration was to determine a reference porosity using one of the two sample slices. The volume of the cavities in the filter cake corresponded to the amount of liquid occupying the voids. Therefore, V_{cav} could be determined by measuring the weight loss (m) when the liquid drains from the cavities by unfreezing of the slice. The slice was placed on a filter paper in a funnel to remove the liquid phase. To avoid the liquid from evaporating within the particles, the filter cake was not dried at high temperatures. Thereby, only the macro pores between the particles but not the internal particle porosity was considered as filter cake porosity. A conversion of m into V_{cav} was performed by considering ρ_f, which was determined using a DMA 4500 density metre (Anton Paar GmbH, Graz, Austria). Accordingly, Equation (4) was modified to

$$\varepsilon = \frac{m}{V_{tot} \cdot \rho_f}. \tag{5}$$

V_{tot} was determined by measuring the side length of a frozen cake slice using a slide gauge.

Parallel to the determination of the reference porosity, the surface roughness of the second slice of the layer was measured. It was placed on a porous medium during unfreezing, which allowed a slow absorption of the liquid to avoid disruption of the cake structure. The surface roughness was analysed by scanning the sample using a VHX-950F digital microscope (Keyence Deutschland GmbH, Neu-Isenburg, Germany) at a magnification of 50× . Gaussian filters (low-pass: 800 μm, high-pass: 2.5 mm) were applied for smoothing and removal of undulations using the microscopes software (VHX-H4M). The data was merged with MATLAB R2018b (The MathWorks, Inc., Natick, USA). Six measurements were recorded from each slice to obtain an average value of the surface roughness.

The small particle size of the fines did not allow the determination of their surface roughness, thus limiting the application of the method to the magnification of the microscope. However, the fine layer is a variable and will be modified or removed in the subsequent experiments. Therefore, the fine layer was excluded from the calibration, and the stamp effect was investigated indirectly via a change in the compression of the four bottom layers.

To obtain porosity values in a wide range for the calibration, filter cakes from the lowest (Fig. 2, point A) to the highest (Fig. 2, point D) compression were produced by varying the volume of the filtrate. A high surface roughness corresponds to a high porosity, and a linear correlation between surface roughness and porosity was observed ($R^2 = 0.97$; $p < 0.001$). The correlation enables the determination of the porosity via a measurement of the surface roughness. This is consistent with similar calibration methods found in the literature (Sakai and Nakamura, 2005; Rebollo et al., 1996) where the measurement was validated using conventional methods.

After the successful calibration, both slices per layer could be used to measure the surface roughness in the following experiments to obtain an average of 12 measurements.

2.3. Modification of filtration tests

To prove the stamp effect, the suspensions were modified to either emphasise or lower the fine-particle-dependent cake compression.

Three different types of malt were used to investigate the dependence of the compression on the raw material. The malts differed in their cytolytic degree of modification and the barley variety in order to obtain different filtration behaviors. As determined in the pre-trials, the malts varied in their flow rate (#1: medium, #2: high, #3: low). Malt 1 with an intermediate flow rate was used for all trials as standard, unless stated otherwise.

An emphasised stamp effect was examined via an oxidation of the suspension where no reducing agent was added compared with the SF-trial. Contrarily, a lower stamp effect was tested by removing the fines by performing wet sieving of the suspension over a sieve (mesh size: 500 μm). The volume of the removed particles was replaced by a filtrate from a previous test, and no modification of the larger particles (>500 μm) occurred.

2.4. Development of filtration technique

After the stamp effect has been proven, a new filtration technique was established to avoid the formation of the blocking layer by the removal of the fine particles.

The technique was based on a standardised suction of the fines prior to filtration from the top of the filter cake. It was applied to both lab-scale filters to study the impact on flow rate and cake porosity

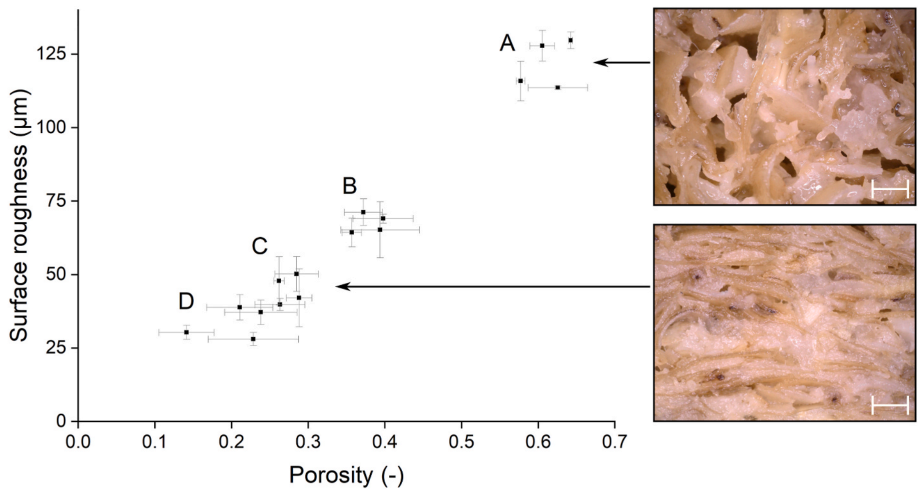

Fig. 2. Surface roughness as a function of the gravimetric reference porosity (left, $R^2 = 0.97$, n = 3). Four states of compression are presented from minimum (A) to maximum compression (D). Only the bottom layers are presented. Representative microscopic images of cake layers (right) show the state of compression before the start of filtration (A) and after the standard filtration test (C). Scale bar represents 1 mm.

compared with the SF-trial without suction. In the first setup (LT-trial, Fig. 1, blue), ~30% of the suspension was removed from the top of the filter after the sedimentation rest using a suction basket (mesh size: 500 µm) connected to the previously described peristaltic pump. The suction basket was placed above the cake to avoid the uptake of coarser particles.

The second setup involved the suction not only from the filter but already from the preceding step in the MT. In this combined suction from MT and LT (MT/LT-trial, Fig. 1, green/blue), ~24% of the suspension was first removed from the MT before transferring to the filter and then additionally ~30% from the LT. For the suction from the MT, a sedimentation rest of 5 min without stirring allowed the coarser particles to settle. The amount of the removed suspension from the MT was replaced by an equal amount of water (78 °C). Filtration was conducted for both setups until the same amount of filtrate was collected as for the SF-trial. Cloudy filtrate was not recirculated back to the filter for the suction trials but was added to the fine-particle suspension instead. Clear filtrate was retained from the suspension by centrifugation (Fig. 1, I) at 4000 rpm for 5 min in a Heraeus Multifuge 4 KR (Thermo Fisher Scientific Inc., Waltham, USA).

2.5. Pilot scale

The new filtration technique was verified on a pilot-scale plant. The effect of the fine-particle removal by suction on the process time, extract yield and filtrate quality was investigated.

The suspension was produced according to the lab scale. Raw material (8.3 kg) was milled on a two-roller mill (Künzel Maschinenbau GmbH, Mainleus, Germany) at a milling gap of 0.8 mm. A 5-min sedimentation rest after transfer to the LT was followed by 5 min of cloudy filtrate recirculation. After collecting the first wort (15 kg), two equal batches of water (sparging) were applied to wash the filter cake until 63 kg of total filtrate was obtained. The filter cake permeability was maintained by standardised raking, as conducted in the practical LT operation. Filtration was performed at constant parameters (flow rate, cloudy filtrate recirculation and sparging), unless stated otherwise. For suction trials, the fine-particle suspension was removed using a suction basket (mesh size: 500 µm) attached to a JP-06 pump (ESSKA.de GmbH, Hamburg, Germany). For the MT/LT-trial, ~43% of the first sparging water was used in the MT prior to the transfer to the LT to replace the volume of the removed fine-particle suspension.

The extract yield and the washable extract (dry matter) in the fines and filter cake were determined according to MEBAK (Mitteleuropäische Brautechnische Analysenkommission) (Miedaner, 2002). The extract was measured using the previously described density metre. The filtrate characteristics (turbidity and photometric iodine value) were determined based on their importance for the subsequent production steps in brewing (e. g. fermentation) and their impact on the product quality (e.g. shelf life). Turbidity was measured using a LabScat turbidimeter (Sigrist-Photometer AG, Ennetbürgen, Switzerland) after cooling the filtrate to room temperature. The photometric iodine value was determined according to MEBAK.

2.6. Statistics

Each filtration test in the lab and pilot scale was conducted in triplicates. The means and standard deviations were calculated. Statistical evaluation was conducted using OriginPro 2019b (OriginLab Corporation, Northampton, USA). Analysis of variance and *t*-test ($p < 0.05$) were employed to determine significant differences between means.

3. Results and discussion

3.1. Proof of the stamp effect and its impact on the cake compression

To identify the stamp effect during filtration, LT-1 was used to trigger cake compression. A sudden decline in the flow rate at 9.1 min (Fig. 3a, point C) was observed, where it decreased from 116.5 g/min (±0.6) to 22.0 g/min (±0.3).

However, this point of decrease is delayed compared with the pressure drop in the filtrate pipe: first, the pressure starts to drop at 6.5 min (Point B), and then, the flow rate decreases at point C. Cake height measurement sheds light on the process (Fig. 3b). Between points A and B, only a small decrease in height by 1.0 cm (compaction rate: 0.15 cm/min) and no change in the flow rate were observed. At point B, high amount of fine particles are deposited on the top of the cake. Consequently, the permeability on the top decreases and the blocking starts. From point B, the filter cake shrinks by 2.4 cm at a high compaction rate of 0.92 cm/min until point C is reached. This indicates the compression of the bottom layer by the stamp effect. No change in the flow rate is observed because the remaining liquid in the bottom layer flows out of the cake without resistance during the compression. This ends at point C, where the rate of pressure drop decreases and a significant decrease in the flow rate occurs. After point C, the low permeability of the top layer impairs the liquid flow, and the filtration rate is determined by the flow resistance of the fine layer. Up to point D, there is only a small

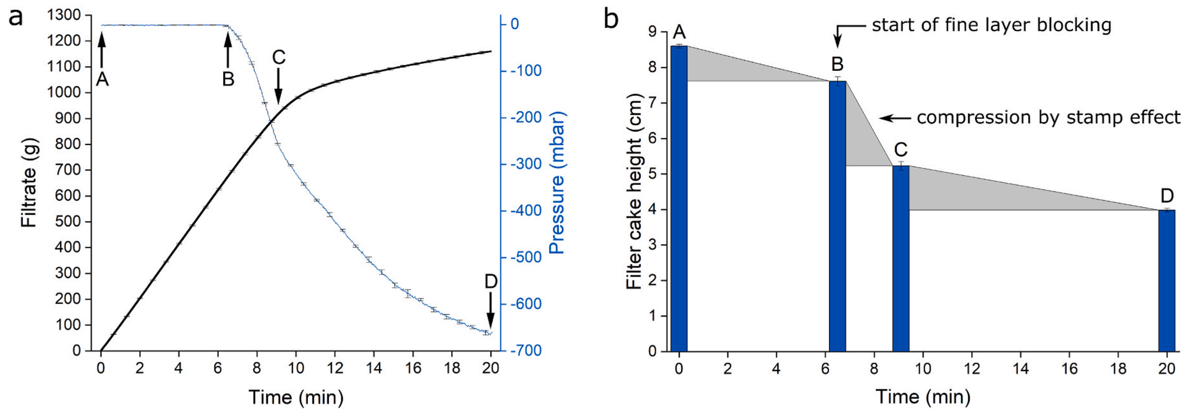

Fig. 3. **a**) Comparison of filtrate mass per time (black) and pressure drop (blue) in the filtrate pipe; **b)** filter cake height (n = 3). Indicated are the start of filtration (A), start of pressure drop (B), turning point of flow rate (C) and end of filtration (D). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

compression of 1.2 cm (compaction rate: 0.11 cm/min) until the maximal compression is reached at the end of filtration.

The cake compression and flow rate reduction observed using LT-1 indicate the influence of the stamp effect on filtration. However, the cake height provides no detailed insights into the change of the porosity during filtration. Therefore, the new method for cake fixation and porosity determination was applied to evaluate the individual cake layers obtained using LT-2.

After the sedimentation rest, the average porosity of the bottom layers 1–4 is 0.623 (±0.002) at the start of filtration (Fig. 4, start filtration). This is in agreement with the porosity value of 0.68 for filter cakes in the LT indicated in the literature (Bühler et al., 1996). During filtration, the average porosity reduces to 0.312 (±0.005) for the SF-trial (Fig. 4, SF). The change in the filter cake porosity can be observed visually (Fig. 2). While cavities in the cake are visible at the start of filtration, the structure is more compressed and cavities are reduced after the SF-trial.

The highest layer (1) for the SF-trial indicates a significant lower porosity compared with layers 2 and 3, which is in contrast to the respective layers at the start of filtration. This is based on the fines that are deposited on top of layer 1 during filtration and indicates the compression based on the stamp effect.

The significant lower porosity of layer 4 compared with layer 3 in the SF-trial is based on the skin effect that occurs next to the filter medium. However, the porosity reduction of layer 4 during filtration is rather low compared with layer 1. This distinguishes the LT operation from other types of cake filtration, in which the porosity decreases based on the skin effect from top to bottom. It indicates that the stamp effect is predominant in lautering compared with the skin effect.

Using various types of malt, the impact of the stamp effect on the filtration of suspensions produced from different raw materials was checked. In addition to malt #1 as in the SF-trial, the flow rates of malts #2 and #3 were evaluated in the filtration tests using LT-1 (Fig. 5).

Similar to the SF-trial, the two other malts exhibit a characteristic reduction in the flow rate after the occurrence of blocking. Compared with the SF-trial (22.6 g/min ±0.3), malt #2 (36 g/min ±1) shows a higher and malt #3 (19 g/min ±1) a lower flow rate. Because the permeability of the fine layer determines the rate of flow, the differences indicate the intensity of the stamp effect for the respective malt types. The higher porosity for malt #2 (0.43 ±0.01) (Fig. 4, malt #2) and the lower value for malt #3 (0.26 ±0.01) (Fig. 4, malt #3) compared with the SF-trial match the difference in the flow rate and, thus, the impact of the stamp effect. In addition, no significant difference was observed between the porosity in all layers for malt #2. This indicates that the stamp effect is less pronounced for this malt, which is in agreement with the higher flow rate and higher average porosity. Malt #3 shows a significant lower porosity for layer 1 compared with layer 2, similar to that observed in the SF-trial. Because there is no significant difference among layers 2–4, the skin effect was not detected at all for this malt. This is a further hint on the predominant impact of the stamp effect, particularly when low flow rates are detected.

The use of different malts revealed that the stamp effect depends on

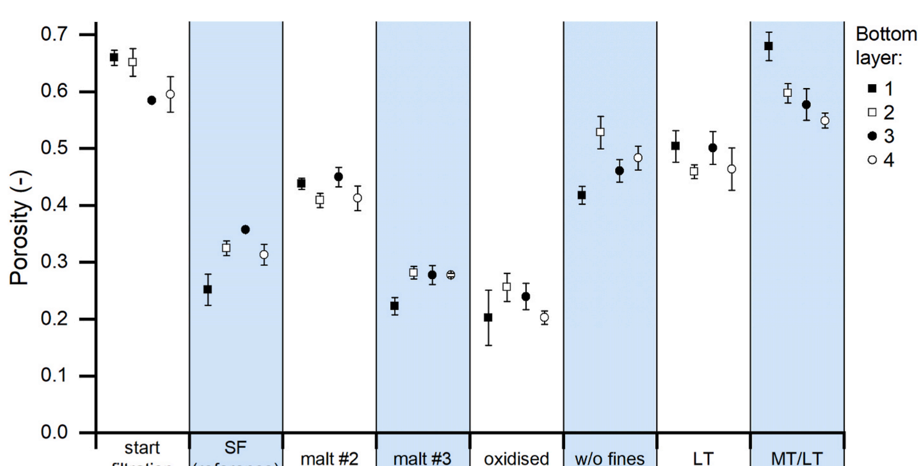

Fig. 4. Porosity of the filter cake layers from 1 (uppermost bottom layer next to the fine layer) to 4 (lowest bottom layer next to the filter medium) for different experiments (n = 3, SF = standard filtration test as reference, LT = suction from lauter tun, MT/LT = combined suction from mash and lauter tun).

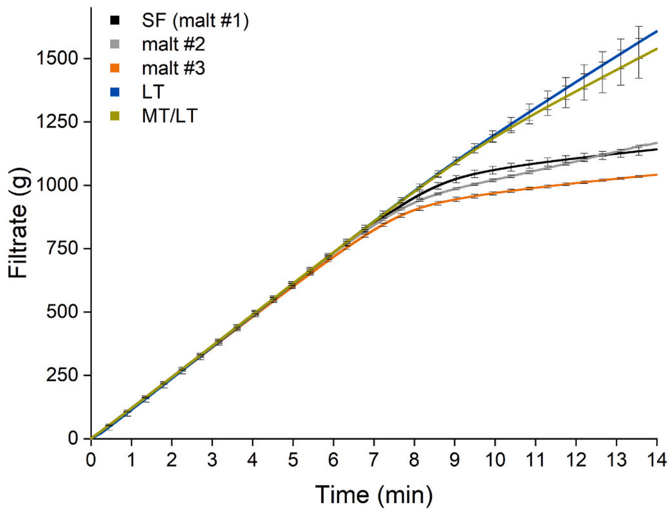

Fig. 5. Filtrate mass per time for standard filtration (SF) test as reference, different malt types (malt #2 and #3) and filtration techniques that include suction from lauter tun (LT) or combined suction from mash and lauter tun (MT/LT) (n = 3).

the raw material. Furthermore, it verifies the method of porosity measurement, which is in agreement with the flow rate when considering filter cakes that are differently produced.

To prove the stamp effect, the fine particles were modified, and the influence on the cake porosity was evaluated. Compared with the SF-trial, an oxidised form of the fine particles results in a lower average porosity of 0.23 (±0.03) (Fig. 4, oxidised). This can be explained by the negative effect of oxidation that decreases the permeability of the fine layer, which results in a more pronounced stamp effect. Conversely, the removal of the fines results in a higher average porosity of 0.47 (±0.01) (Fig. 4, w/o fines). The low porosity of layer 1 in this test indicates that fine particles were not completely removed and a low stamp effect is still present.

Considering the modifications of the SF-trial, it can be concluded that the fine layer has the largest impact on the cake compression and serves as proof of the stamp effect. The skin effect is less pronounced compared with the stamp effect.

The difference between stamp and skin effect is summarized in a scheme (Fig. 6) that was created on the basis of the present filtration experiments. In the case of the skin effect, the lowest porosity can be observed near the filter medium. In contrast to this, the porosity at the top of the filter cake is lowest for the stamp effect. This leads to an overall lower cake porosity compared to the skin effect.

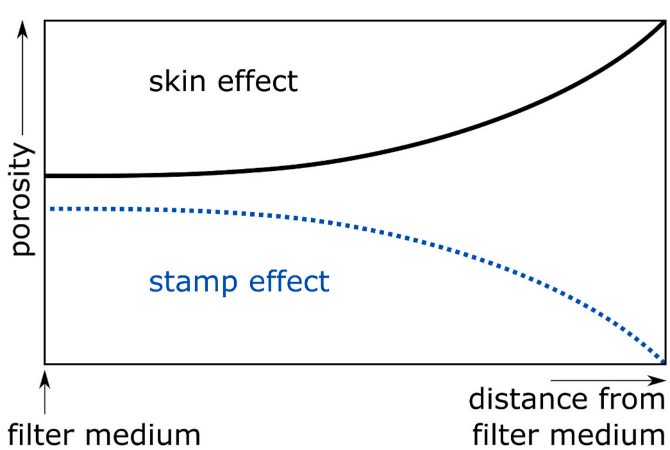

Fig. 6. Representation of the differences in the porosity of the filter cake for the stamp (dotted line) and skin (solid line) effect at the end of the filtration process.

3.2. *Prevention of the stamp effect*

After the stamp effect has been proven, a technique to avoid it in the practical process was developed. The removal of the fines by suction from the LT (Fig. 1, F, LT-trial) or by combined suction from the MT and LT (Fig. 1, G, MT/LT-trial) significantly reduced the cake compression. A higher average porosity of 0.48 (±0.03) for the LT-trial (Fig. 4, LT) and 0.60 (±0.02) for the MT/LT-trial (Fig. 4, MT/LT) proved the success of the technique. Because layer 1 demonstrates a similar or even higher porosity compared with layers 2–4 for both trials, it can be concluded that the stamp effect is reduced. For the MT/LT-trial, the average porosity is similar to the initial cake porosity (Fig. 4, start filtration). In addition, the porosity increases for the MT/LT-trial from the bottom to top. Because this is typical for common filtration processes, it indicates that the skin effect prevails and that the stamp effect is no longer present.

Two effects are responsible for the lower compression of the cake for both techniques. First, based on a lower amount of fines on top of the filter cake, the stamp effect was significantly reduced. Second, the filtrate was recovered directly from the removed suspension via centrifugation. Consequently, the volume that had to be filtered through the cake was reduced, which results in a decreased compression.

The impact of a reduced stamp effect using the new techniques was verified by investigating the flow rate and the blocking potential (Fig. 5). For the LT-trial, as well as the MT/LT-trial, the characteristic blocking of the cake is significantly lower, which can be observed in a high flow rate of 104 g/min (±12) for the LT-trial and 90 g/min (±16) for the MT/LT-trial. This serves as a proof that the stamp effect can be avoided by removing the fines in the practical filtration process. No significant difference was observed between the flow rate of the LT- and MT/LT-trial, which means that suction from the top of the LT is already sufficient to avoid cake blocking.

3.3. *Verification in pilot scale*

The technique was verified on a pilot-scale plant to investigate the effects on the process time, yield and product quality.

Compared with the SF-trial, the time is reduced by 45% for the LT-trial and by 46% for the MT/LT-trial (Table 1). The reduction is mainly based on the lack of the first wort filtration time, which constitutes 40% of the total process time. Considering the occupation time of the LT only, the time saving for suction from MT/LT is even 55% compared with the SF-trial, because removal of a part of the suspension was already conducted in the MT. Consequently, a part of the sparging water was already added in the MT, which reduced the required time for the first sparging in the LT. Based on a higher cake porosity, the flow rate during sparging was increased, which further reduced the time. As a side

Table 1
Overview of the process time for the pilot-scale trials of the standard filtration (SF) test compared with the suction from the lauter tun (LT) and combined suction from mash and lauter tun (MT/LT) (n = 3).

	SF	LT	MT/LT
Sedimentation rest MT (min)	–	–	5
Suction MT (min)	–	–	3
Sedimentation rest LT (min)	5	5	5
Suction LT (min)	–	5	5
Cloudy filtrate recirculation (min)	5	5	5
First wort (min)	36 (±3)	–	–
First sparging (min)	26.3 (±0.5)	17 (±2)	9.0 (±0.8)
Second sparging (min)	16.3 (±0.5)	17 (±1)	15.7 (±0.5)
Overall process duration (min)	88.6	49	47.7
Overall time saving (%)	–	45	46
Time saving in LT (%)	–	45	55

effect, deep cuts during raking could be avoided in the suction trials. In addition, there was no need to recirculate the cloudy filtrate back to the filter for both suction trials. Instead, it was added to the fine-particle suspension that was centrifuged, reducing the amount of fluid that had to be filtered through the cake. The centrifugation of the fine-particle suspension was executed parallel to the LT operation and, therefore, had no effect on the total process time.

The fine particles recovered after centrifugation were not washed with water during sparging. Consequently, the remaining washable extract (dry matter) in the fine particles was significantly higher for the LT-trial (53.9% ±0.9%) and MT/LT-trial (52% ±1%) compared with the remaining extract in the entire filter cake for the SF-trial (5.0% ±0.4%). However, a comparison of the extract yield of the total process reveals no significant differences for the suction trials compared with the SF-trial (Table 2). This is due to the small amount of fine particles removed from the suspension. Compared with the initial amount of raw material, only 0.33% (±0.06%) of fine-particle dry matter was removed for the LT-trial and 0.40% (±0.06%) for the MT/LT-trial. Therefore, the extract loss based on the fines has no significant influence on the total extract yield in relation to the entire filter cake.

The turbidity of the first wort is significantly reduced for the LT-trial and in an even higher degree for the MT/LT-trial compared with the SF-trial (Table 2). This is due to the more efficient separation of the particles based on the centrifugation. For the total filtrate, no significant difference was observed for the LT-trial compared with the SF-trial, which can be a side effect of the higher cake porosity. Because the amount of centrifuged suspension is the highest in the MT/LT-trial, the turbidity is the lowest for first wort and total filtrate.

No significant difference for the photometric iodine value was observed for the total wort of the LT-trial compared with the SF-trial (Table 2). For the MT/LT-trial, a significant lower iodine value was obtained.

Because the critical filtrate characteristics (turbidity and iodine value) of the product are either constant or even lower for the suction trials compared with the SF-trial, no negative effects on the subsequent process steps or product quality are to be expected for the new filtration techniques.

4. Conclusions

A new method for cake fixation and porosity determination was developed to prove the stamp effect in the LT operation. The method was successfully implemented. Freezing of the cake structure enabled sampling at different time of filtration and from individual cake layers. This approach enabled the consideration of the special requirements of the inhomogeneous filter cake in the LT. In combination with the optical porosity determination via a measurement of the surface roughness, an investigation of the stamp effect was successful.

Fine particles on the top of the filter cake were found to be responsible for the stamp effect in the LT, which resulted in the compression of the bottom layer and, thus, reduction of the overall flow rate. The stamp effect turned out to be the predominant factor influencing the filtration behaviour. Contrary to that observed in other types of cake filtration, the skin effect next to the filter medium has only a minor impact in the LT. However, factors such as the raw material or the influence of oxidation in the practical process determine the strength of the stamp effect.

Up to now, there are no approaches to avoid the stamp effect in the practical LT operation. Therefore, an alternative filtration technique was developed based on the new findings. The fines were removed from the filter prior to the filtration process, which resulted in a lower cake compression and higher flow rate. A transfer to the pilot scale revealed that a significant reduction in the process time is possible without negative effects on the yield or filtrate quality. The verification of the filtration technique serves as a proof that the prevention of the stamp effect improves the process.

The present work serves as a suggestion for an industrial scale-up of the technique. A possible implementation in the brewery could involve the suction of the suspension after the mash transfer using an opening on the side of the LT above the bottom layer of the filter cake. After transferring the suspension to a storage tank, a disc stack centrifuge could be used to clarify the wort. The filtrate could then be transferred directly to the wort kettle for the subsequent production step.

However, one has to consider that the new technique includes an additional process because the filtrate has to be recovered from the fine-particle suspension. In an industrial scale-up, it must be checked if higher acquisition and operating costs are weighed up by a reduced process time.

Table 2
Overview of extract yield, turbidity and iodine value for the pilot-scale trials of the standard filtration (SF) test compared with the suction from the lauter tun (LT) and combined suction from mash and lauter tun (MT/LT) (n = 3).

	SF	LT	MT/LT
Extract yield (%)	68.6 (±0.9)	67.6 (±0.5)	68.0 (±0.6)
First wort turbidity 25° (EBC)	253 (±21)	145 (±14)	104 (±6)
Total filtrate turbidity 25° (EBC)	155 (±15)	186 (±19)	114.7 (±0.9)
Iodine value (−)	0.31 (±0.04)	0.22 (±0.03)	0.18 (±0.01)

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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References

Alles, C.M., Anlauf, H., 2003. Filtration mit kompressiblen Kuchen: Effiziente Konzepte für eine anspruchsvolle Trennaufgabe. Chem. Ing. Tech. 75 (9), 1221–1230. https://doi.org/10.1002/cite.200303268.

Barrett, J., Clapperton, J.F., Divers, D.M., Rennie, H., 1973. Factors affecting wort separation. J. Inst. Brew. 79 (5), 407–413. https://doi.org/10.1002/j.2050-0416.1973.tb03558.x.

Bühler, T.M., McKechnie, M.T., Wakeman, R.J., 1996. A model describing the lautering process. Monatsschrift für Brauwissenschaft 7/8, 226–233.

Bühler, T.M., McKechnie, M.T., Wakeman, R.J., 1996. Temperature induced particle aggregation in mashing and its effect on filtration performance. Food Bioprod. Process. 74 (4), 207–211. https://doi.org/10.1205/096030896531208.

Engstle, J., Briesen, H., Först, P., 2017. Mash separation in the lauter tun - a particle size dependent separation process. Brew. Sci. 70, 26–30.

Engstle, J., Reichhardt, N., Först, P., 2015. Filter cake resistance of horizontal filter layers of lautering filter cakes. MBAA Tech Q 52 (2), 29–35. https://doi.org/10.1094/tq-52-2-0405-01.

Engstle, J., Ueffing, H., Först, P., 2015. Bildanalytische Überwachung des Läutervorganges. Brauwelt 36, 1040–1042.

Hennemann, M., Gastl, M., Becker, T., 2019. Inhomogeneity in the lauter tun: a chromatographic view. Eur. Food Res. Technol. 245, 521–533. https://doi.org/10.1007/s00217-018-03226-4.

La Heij, E.J., Kerkhof, P.J.A.M., Kopinga, K., Pel, L., 1996. Determining porosity profiles during filtration and expression of sewage sludge by NMR imaging. AIChE J. 42 (4), 953–959. https://doi.org/10.1002/aic.690420408.

Martin, W.D., Putman, B.J., Kaye, N.B., 2013. Using image analysis to measure the porosity distribution of a porous pavement. Construct. Build. Mater. 48, 210–217. https://doi.org/10.1016/j.conbuildmat.2013.06.093.

Mathmann, K., Kuhn, M., Briesen, H., 2014. Application of micro-computed tomography in food and beverage technology using the examples of textured vegetable protein and filtration steps in the brewing process. Wels, Österreich.

Miedaner, H., 2002. Brautechnische Analysenmethoden. Methodensammlung der Mitteleuropäischen Brautechnischen Analysenkommission 2 (Freising-Weihenstephan).
Rebollo, M.A., Hogert, E.N., Albano, J., Raffo, C.A., Gaggioli, N.G., 1996. Correlation between rougness and porosity in rocks. Optic Laser. Technol. 28 (1), 21–23. https://doi.org/10.1016/0030-3992(95)00051-8.
Sakai, T., Nakamura, A.M., 2005. Quantification of porosity and surface roughness in laboratory measurements of the bidirectional reflectance of asteroid surface analogues. Earth Planets Space 57, 71–76. https://doi.org/10.1186/BF03351807.
Tiller, F.M., Green, T.C., 1973. Role of porosity in filtration IX Skin effect with highly compressible materials. AIChE J. 19 (6), 1266–1269. https://doi.org/10.1002/aic.690190633.
Tippmann, J., Voigt, J., Sommer, K., 2011. Measuring particle size distribution of mash with laser diffraction to evaluate the process success. Brew. Sci. 64, 13–21.