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Characterizing the Filtration Behavior of Hop Particles for Efficient Dry Hopping Methods

The market of craft beer has been constantly growing in the last few years. Within the craft beer movement, dry hopped beers are of preeminent importance. In order to achieve the pronounced aroma profile desired in these beverages, hops, mostly in the form of pellets, are introduced into the fermentation or maturation tank. Consequently, the added solid particles have to be removed after the aroma extraction. Since the available separation equipment in most breweries is not optimal for this task, a considerable amount of product is often lost in the process. Therefore, cake-forming filtration is explored as a promising alternative for the currently used solid-liquid separation techniques. This study reports data regarding the properties of hop filter cakes and the relevant operational parameters for the separation of hop particles from beer. These findings may directly help breweries reduce the duration of dry hopping while increasing the product yield.

Descriptors: dry hopping, hop pellets, separation, cake filtration, permeability

1 Introduction

Dry hopping is a method that is used to produce beers that are characterized by outstanding bitterness and various aromas. Although this technique has been known for many years, it had been neglected before the craft beer movement in the USA put it on the map again.

To brew beers with distinctive aroma profiles, hops are dosed after the wort is cooled down, as opposed to late hopping [1]. Pellets are the most widely used hop product in this case because they are easier to store and administer than the alternatives [2]. The resulting beer has become so popular that the number of craft breweries is constantly on the rise, as are the sales figures of craft beers. Dry hopped craft beers are, however, normally more expensive than major domestic beers. Besides the material costs, the process involves additional challenges for the brewer; in particular, the volume of solids in the beer grows remarkably. The dosed particles have to be separated afterwards, which brings about risks such as product loss (due to adsorption, oxidation, or contamination) or even beer quality deviations [1, 3–5]. In order to minimize product loss, new process strategies are needed. To this end, the following hypothesis is addressed: applying a cake filtration process to separate the solids added to the beer improves the product yield while reducing the duration of the dry hopping process. It is thus expected that the aroma extraction and the separation can be

conducted simultaneously and that the resulting filter cake does not trap considerable amounts of product.

Dry hopping is known to be a process step that is based on mass transfer. As such, research has been focused on the extraction behavior of aromatic substances [6–9] as well as their influence on the taste and stability of the product [10–13]. Nonetheless, open questions remain regarding the necessary unit operations for an efficient process. The principles of the method are well known, but it still lacks a scientific basis, especially when it comes to novel separation processes.

In order to gain some insights into this question, it is necessary to break dry hopping down into smaller steps. The properties of the method with pellets can be studied as represented in table 1.

Mass transfer constantly occurs, as long as there is contact between the phases. This can be a rather simplified way to classify the prevailing phenomena, but the main variables implied have already been investigated. Engstle et al. [14, 15] started by determining the end volume of pellet beds of various types. Their results suggested that pellets made of hops with a low alpha acids

Table 1 Process stages during dry hopping and relevant associated material properties

Stage	Properties	Mass transfer
Swelling	Swelling volume Disintegration time	
Dispersion	Particle size distribution	
Sedimentation	Velocity Homogeneity	
Filtration	Cake resistance Compressibility	
Permeation	Permeability Porosity	

<https://doi.org/10.23763/BrSc18-16bandelt>

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content tend to swell more. Consequently, such hop pellets are expected to have a larger surface area and to therefore be more convenient for hopping. The hop variety, however, showed no influence on the disintegration time. The most influential variable for disintegration time was temperature, since elevated temperatures reduce the hardness of the waxy substances in hops. It was shown that an increase of 20 °C reduced the time by at least 85 %. In addition, it was shown that higher alcohol contents and lower pH values prolong the disintegration, making water a more suitable solvent than beer. The authors also presented results regarding the sedimentation velocity of the particles and their size distribution, concluding that the concentration of solids has a positive influence on the homogeneity of the sedimentation bulk but that convection is a better method to promote hopping.

In studies such as the one conducted by *Banke and Pillmeier* [3], alternatives have been discussed for making the aroma extraction more efficient, thereby confirming the validity of the principles described above. Further, they showed that using a targeted engineering solution to improve the dry hopping method can double the yield of aroma substances in the product within 1–3 h. However, these authors only briefly addressed the posterior separation of the particles.

In order to fill in the knowledge gap between pellet dispersion and product finishing, this work focuses on the process parameters when separating hop particles through cake filtration. From a general standpoint, the most relevant associated variables are filter cake resistance and permeability. Therefore, this analysis is based on the description of flow through porous media with Darcy's law:

$$\dot{V} = \frac{\Delta p \cdot A \cdot k}{\eta \cdot h}. \quad (\text{Eq. 1})$$

Darcy's law states the proportionality between the volumetric flow rate \dot{V} and the pressure difference Δp . \dot{V} also depends on the cross-sectional area A , the permeability k , the filtrate dynamic viscosity η , and the height of the porous medium h . Permeability is characteristic for the given porous structure, and it can be estimated experimentally.

2 Materials and Methods

The experiments were conducted with hop pellets of type 90 obtained from the hop variety Cascade (Simon H. Steiner, Hopfen, GmbH). This variety contains 16.5 % total resin as reported by the manufacturer, which contributes to the compressibility of the system [14]. To discard possible influences caused by the hop type, the permeability experiments were repeated using pellets made of the variety Hallertauer Tradition (15.6 % total resin), whose behavior was found to be identical.

To assess the disintegration behavior of a hop pellet, it was submerged in an Erlenmeyer flask filled with purified water. The flask was set in front of a webcam, which was programmed to take a picture each minute until no changes could be observed. This procedure was also applied when monitoring the sedimentation of hop particles (50 g of pellets dispersed in 2 L of purified water)

in an Imhoff Cone and a cylindrical sedimentation column (10 cm diameter, 12.7 mm outlet). The concentration of the suspension in the sedimentation column was subsequently varied between 5 and 10 m% in order to determine the point above which the suspension loses its capacity to flow. Each concentration was prepared and allowed to settle overnight. The suspension was then homogenized, and the bottom valve of the column was opened to start the flow.

The specific volume of the pellets was derived from their density, which was determined using a gas pycnometer (AccuPyc 1330, Micromeritics GmbH), and the particle size distribution (PSD) of the suspension was analyzed using the laser diffraction setup Helos/KR (Sympatec GmbH). The Helos samples were suspended in water and uniformly dispersed in the setup using the Helos dispersion unit Quixel.

The filtration experiments were conducted using a stand designed and built by Banke process solutions GmbH & Co. KG following the norm of the Association of German Engineers (Verein Deutscher Ingenieure) [16]. The equipment comprised two separate glass housings to disperse and filter the suspensions under pressure. The first housing was equipped with a stirrer and had an internal diameter of 130 mm and a volume capacity of 2 L, whereas the second housing had an internal diameter of 80 mm, which is equivalent to a filtering area of 50.27 cm². The ambient temperature was maintained at 20 °C using a Julabo GmbH F30 VC/3 thermostat. A steel sieve with a retention capacity of 55 µm was used as the filter medium. The filtrate mass was automatically measured using a Kern & Sohn GmbH KB 10000-1N precision scale. The data was processed using a Matlab (The MathWorks, Inc.) code. A schematic of the equipment is illustrated in figure 1.

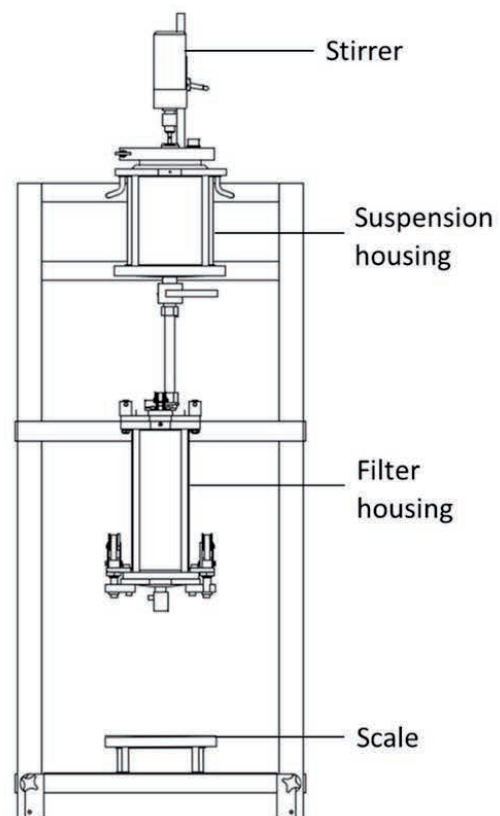


Fig. 1 Experimental filtration equipment

Table 2 Tested parameters during the experiment series

Series	Temperature [°C]	Pressure [bar]	Yeast cells [$10^6/\text{mL}$]
Filtration	20	0.2	0
Water permeation	[5; 20; 85]	[0.2; 0.5; 1.0; 1.5; 2.0; 2.5; 3.0]	0
Beer permeation	5	0.6	[0; 2; 5; 15; 30]

The standard filtration curve was thus obtained for the system under a pressure of 0.2 bar. Strong cake compression during filtration was avoided using this comparatively low pressure. All the experiments were performed three times, with the results represented as the arithmetic mean value. The error bars indicate confidence intervals of 95 %.

Each suspension was prepared using distilled water at a final volume of 1.5 L. The pellets were submerged in water before suspension according to the swelling procedure. The sample quantity was determined on the basis of a cake height of 2.7 cm (20 g) to achieve filtration times of about 10 min. The sample was transferred to the suspension housing after configuring its stirrer to maintain homogeneity. Subsequently, the valve to the filter housing was opened, the system was pressurized, and the filtration and data recording were initiated.

The filtration data (accumulated filtrate mass over time) was linearized in the form of $(\text{time}) \cdot (\text{accumulated filtrate volume})^{-1}$ over the same volume to obtain the relative filter cake resistance α_K and the flow resistance of the filter medium R_M according to:

$$\frac{t}{V} = \frac{k \cdot \eta \cdot \alpha_K}{2 \cdot A^2 \cdot \Delta p} \cdot V + \frac{R_M \cdot \eta}{A \cdot \Delta p}, \quad (\text{Eq. 2})$$

which is derived from equation 1. Here, t is the filtration time, V is the filtrate volume, k is a concentration constant calculated from the cake thickness, η is the fluid viscosity, A is the filter area, and Δp is the pressure difference.

The permeability experiments were conducted in a similar fashion. They were performed using a filtration cell with an internal diameter of 66 mm and a volume capacity of 630 mL. The pellets were swollen in purified water at room temperature, and the suspension was then brought to the temperature of interest. The hop particles were separated as before to form the filter cake, whose permeability was then determined by refilling the cell with water at the desired temperature, pressurizing the system at the desired pressure, and letting the liquid flow through the cake. The permeate mass was measured using the same precision scale as before.

In order to determine the influence of yeast cells on the permeability of the hop filter cakes, a further experiment series was performed according to the previous procedure. This time, the pellets were swollen in filtered lager beer and a determined concentration of yeast cells was added (BrewMasters Pilsner Style; Erbslöh Geisenheim AG) before determining the permeability. All the experimental

parameters are summarized in table 2.

3 Results and Discussion

The results of this work focus on the properties of the suspended solid particles and on their separation behavior. To do so, it is important to consider the phenomena that occur beforehand. The swelling time of the pellets was set to 2 h, since changes in the particles are no longer observable after this duration. During this period, the liquid enters the pores of the pellet and weakens the cohesion among the particles. The coarse agglomerates are separated first, and the primary hop particles of the pellets start swelling next, thereby greatly increasing their specific surface. Pellet disintegration is illustrated in figure 2, alongside a scanning electron microscope (SEM) image of the primary particles.

Figure 2 shows the substantial change in the volume of a hop pellet during disintegration and swelling. While the specific volume of the pellets ranges from 0.8 to 1.0 mL/g, the swelling volumes reported by Engstle et al. [14] are at least eight times this value. However, the actual volume depends on the local compression force along the cake height of hop particles [17]. As shown by the SEM image, the primary particles resemble platelets, a type of particle that can be easily compressed depending on its stiffness. If the pressure is low, platelets can maintain a structure that is comparable to a house of cards. At higher pressures, the structure collapses and becomes much less porous [18]. On this account, the larger the hop mass, the denser the structure and the smaller its volume. This explains why swelling factors of 5.6 have also been reported. If a final value of 11 mL/g is assumed and beer is heavily dry-hopped with 3 m% pellets, then the column can occupy around 30 % of the tank's space. This result clarifies the low yield in craft breweries that separate the sediment by letting it off.

The corresponding volumetric PSD was analyzed and is presented in figure 3 in a normalized differential (q_3) and accumulated (Q_3) form.

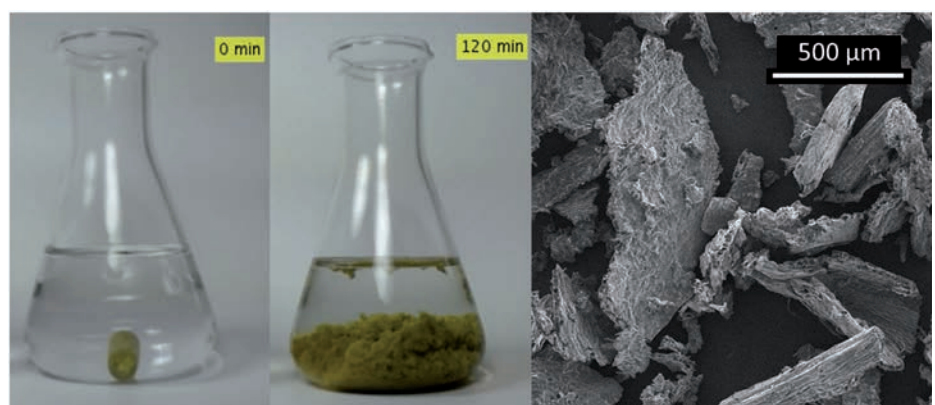


Fig. 2 Swelling of a hop pellet in water at 20 °C and a SEM image of its primary particles

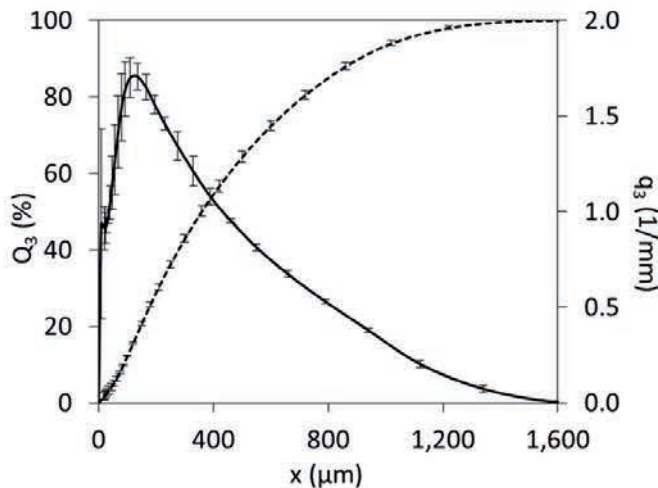


Fig. 3 PSD of hop particles. Q_3 (dashed line) and q_3 (solid line)

The suspended pellets become a polydisperse system. The sample shows a broad PSD with a marked modal value at approximately $135 \mu\text{m}$ and an $x_{50,3}$ value of approximately $360 \mu\text{m}$. Using equation 3, the relative PSD span S was estimated to be 2.25.

$$S = \frac{x_{90,3} - x_{10,3}}{x_{50,3}} \quad (\text{Eq. 3})$$

Since the pellet material is biogenic, it consists of various substances and is therefore relatively complex. The error bars below $400 \mu\text{m}$ are indicative of such complexity.

The Q_3 curve in figure 3 is comparable to the ones already published [14]. This time, however, the sample was stirred for 5 min instead of 2 h after swelling, so the PSD values reported here are higher. Nevertheless, the specific surface of the most frequently occurring particles in figure 3 is considerable and is estimated to be greater than $40,000 \text{ m}^{-1}$. Large surface areas favor mass transfer, which is why swelling and maintaining a constant suspension state of the primary particles should be guaranteed.

If the particles are not kept suspended, they will be able to sediment rapidly and build inhomogeneous cakes depending on the concentration, as has already been reported. This becomes observable when using sedimentation columns or Imhoff Cones (Fig. 4). Different mass concentrations were tested in the column and it could be observed that, in a 9 m% suspension, the amount of liquid is no longer sufficient for the suspension to flow out of it. This concentration, however, is far removed from those encountered in the beer industry.

The sediment inside the column of figure 4 shows a layered structure caused by the broad PSD (Fig. 3). The upper layer exhibits smaller particle sizes owing to the lower settling velocities [19]. This phenomenon is observable not only here but also during lautering [20] and can be described for single

spherical particles in laminar flow with Stokes equation (Eq. 4).

$$v = \frac{d^2 \cdot g \cdot \Delta\rho}{18 \cdot \eta} \quad (\text{Eq. 4})$$

This terminal sedimentation velocity v is thus directly proportional to the square of the particle diameter d . Other factors are the gravitational acceleration g , the density difference between the suspended particle and the surrounding fluid $\Delta\rho$, and the filtrate viscosity η .

The sedimentation of the coarse particles in the Imhoff Cone occurs during the first 10 min, while the fine particles continue settling during the next 20 min. After that time, the inhomogeneous sediment compresses and consolidates. Given these observations, the formation of a filter cake would therefore be more favorable. As in a mash filter, the liquid flow guarantees a more homogeneous and permeable structure, which translates to less pressure loss and faster extraction [21].

The analyses of the hop cakes during filtration and permeation allowed to gather relevant data for the operational design of the process. The filtrate mass as a function of time displayed the expected behavior with a high degree of reproducibility. Linearized data was utilized to determine the coefficients of the linear function. The numerical results of this study (Tab. 3) were derived from the coefficients that were calculated using the linear function.

Figure 5 shows the filtration runs with suspended hop pellets at 0.2 bar and their linearization. Very high reproducibility was achieved for all the experiments, as indicated by the small error bars. The corresponding numerical results are presented in table 3.

Table 3 Medium resistance and relative cake resistance from linearized data

Coefficient	Value
$\alpha_K [\text{m}^{-2}]$	2.968×10^{12}
$R_M [\text{m}^{-1}]$	3.259×10^9
$R^2 [-]$	0.9997

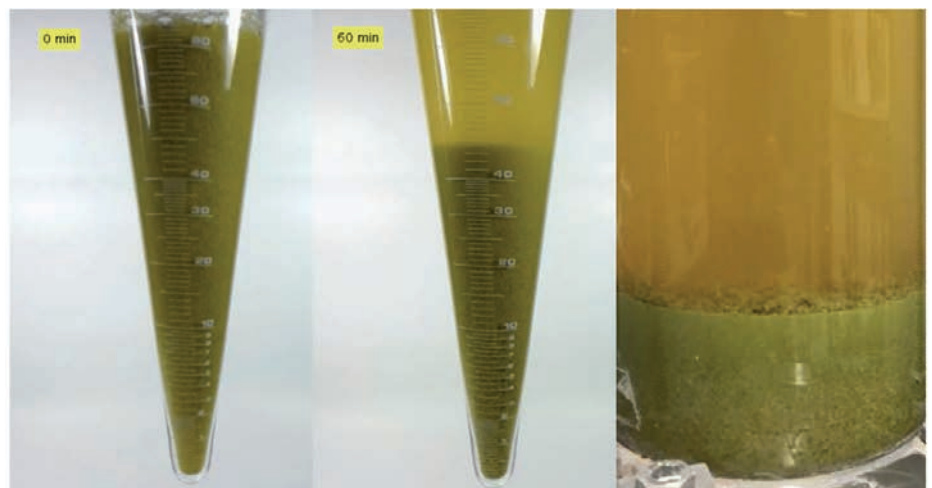


Fig. 4 Hop particles in an Imhoff Cone and in a sedimentation column

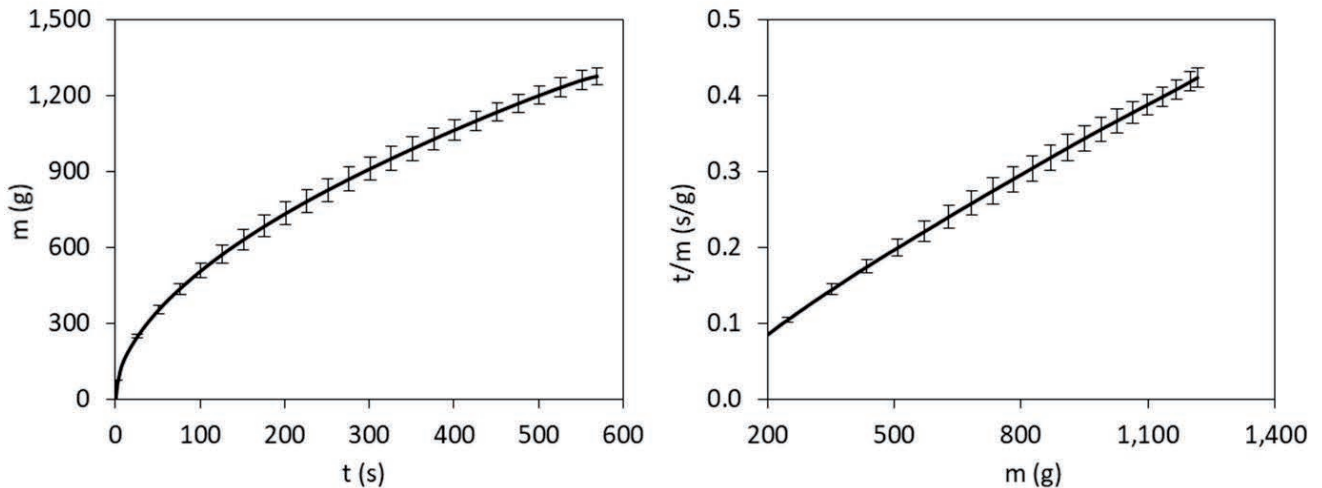


Fig. 5 Filtrate mass over time and linearization of the filtration curve

The coefficient of determination R^2 further confirms the validity of the linear regression. While filtering, R_M was three orders of magnitude lower than α_k . Following the VDI's classification for flow resistance [16], the system can be considered "easily separable" (below $1.0 \times 10^{13} \text{ m}^{-2}$) at low pressures.

Once the cake is formed, the liquid may continue flowing through it until sufficient aroma extraction is achieved. In order to characterize this process step, the permeability of the cakes was determined under different temperatures and pressures. Both parameters were identified as very influential factors on the flow: experiments at low temperatures and pressures exhibited permeabilities up to one hundred times higher than the permeabilities at high temperatures and pressures. These results are summarized in figure 6.

The highest permeability value ($5.0 \times 10^{-12} \text{ m}^2$) was achieved at 0.2 bar and 5 °C. If the temperature or the pressure is increased

above these values, the function will exhibit a quick decline. It is clear that cold-stage separation at low pressures is favorable. Higher pressures cause the solid structure to collapse and become denser, as discussed above. The observed exponential proportionality is a sign of a compressible filter cake. The effect of the temperature was also observed by Engstle et al. [14] while measuring the disintegration time of the pellets. Even though the liquid's viscosity is lower in the hot stage, the cake becomes much denser. This is likely due to the fluidization of the resins that hold the agglomerates together. Under such conditions, more primary particles are released, which build a less porous cake. A comparison of microscope images of cold and hot suspensions (Fig. 7) confirms this.

This effect of primary particle release also applies when further fine particles are present in the suspension, such as yeast cells. Given their smaller size, they can penetrate the filter cake, are separated subsequently by the mechanism of depth filtration, and reduce the porosity of the filter cake. The quantity of the separated cells was found to be correlated with the resulting permeability of the cake (Fig. 8).

The proportion between the cake permeability and the quantity of yeast cells in the suspension can be considered to be linear. A reduction of permeability of up to 40 % was observed in the concentration range of 0–30 million cells/mL. This proves that aroma extraction using unfiltered beers could be very problematic in the industry.

Future work should aim to develop the presented separation approach further and to understand the aroma extraction kinetics better. A plausible process strategy, which can enhance the permeability of filter cakes using common packing materials, has been proposed by *Bandelt Riess* et al. [22] and can potentially counteract the yeast issue.

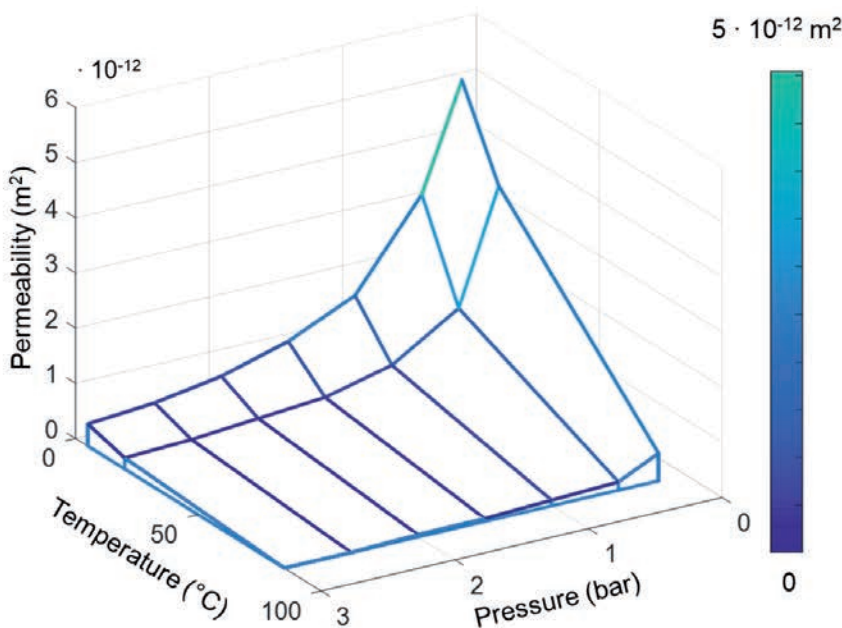


Fig. 6 Permeability of a hop filter cake as a function of temperature and pressure

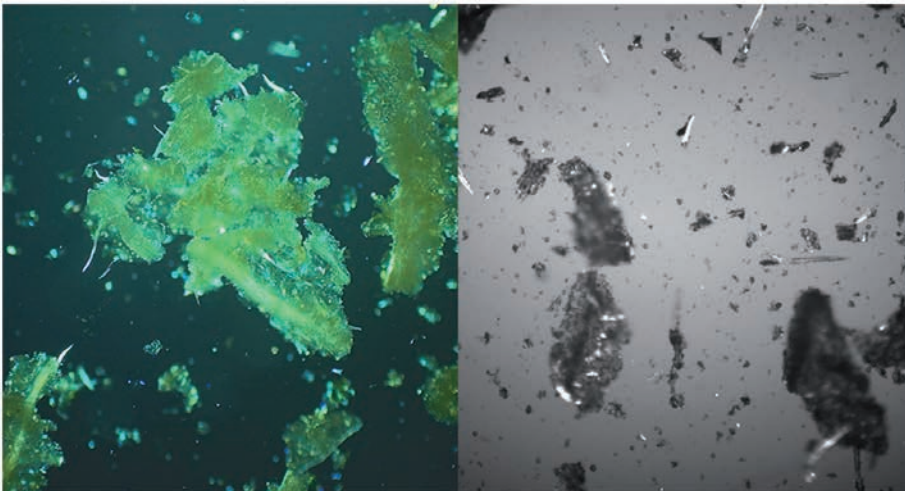


Fig. 7 Hop particles at 25 °C (left) and 80 °C (right)

result also supports a combined filtration-extraction approach. Nevertheless, if yeast cells are present, the pressure drop rises, so the operational parameters might need to be adjusted. The presented data has already been successfully used in the development of hopping devices present in the market.

Acknowledgments

The authors would like to thank Banke process solutions GmbH & Co. KG for their appreciable cooperation during this research project, which was supported by the German Ministry of Economics and Technology (via ZIM Project ZF 4025001SK5).

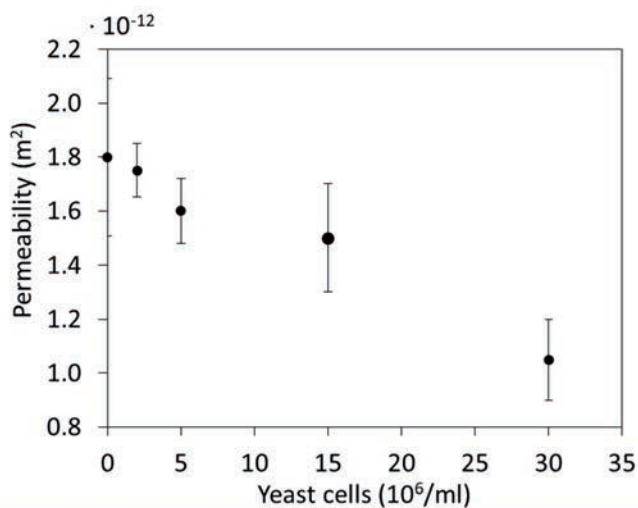


Fig. 8 Permeability of hop cakes as a function of the quantity of yeast cells in the suspension

4 Conclusion

The effects of a filtration approach on dry hopping were studied by means of standard filtration experiments. In this investigation, the dispersion and separation of hop particles after dry hopping were analyzed. The influence of several factors was discussed in order to derive science-based process strategies.

The considerable swelling volume of hop pellets is favorable for aroma extraction due to the surface area increase of the primary particles, which can be dispersed after the swelling with minimal energy input. Swelling of the pellets at higher temperatures can significantly accelerate this step as well as disintegration. Other solvent properties, such as low pH or the presence of alcohol, exhibit a rather negative influence, making brewing water a more suitable solvent for aroma extraction than beer. Hop particles can be classified as “easily separable”. However, performing filtration at higher temperatures can reduce the permeability remarkably, thus justifying cold extraction. Given the compressibility of the hop filter cake, low operating pressures are also beneficial. The homogeneity of the cake is crucial for a low pressure drop; this

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Received 31 August 2018, accepted 7 October 2018