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Increasing Wort Flow by Flocculation of Fine Particle Fractions

As lautering is often considered the bottleneck of the whole brewing process, measures to accelerate this operation are desired. The effect of flocculants on the lautering performance was investigated. Several clarifying agents were analyzed in jar-tests and dosed to the mash at different concentrations. As a result, significant lautering time savings with slight effects on the wort extract were achieved. These findings might directly help breweries to improve their lautering processes and foster further research on the topic.

Descriptors: lautering, mash separation, flocculation, agglomeration, sedimentation, cake filtration

1 Introduction

The mash filtration is the first solid-liquid separation of the brewing process. Small and medium-sized breweries, as well as breweries with a relatively large product diversity, mainly use lauter tuns to obtain the wort from the mash [1]. This process step often is very time-consuming and the lautering performance is very difficult to predict [2]. The remaining spent grain filter cake is highly inhomogeneous and consists of a layered structure [3], which can become a particular obstacle for wort flow [4]. The top layer, the Oberteig, composed of fine particles with sizes smaller than 500 μm , exhibits a great filter cake resistance, which slows down the filtration, and consequently delays production time [5]. In order to optimize this, new process strategies are desirable and the following hypothesis is addressed: flocculating the fine particles of the Oberteig can shorten the lautering duration and have a positive effect on the brewhouse yield. It is thus expected that enlarged particles will be able to sediment faster and build a more permeable spent grain cake, considering that the performance of common cake filtration processes depends on the filter cake's properties [6].

Lautering not only comprises filtration but also a preceding sedimentation [7]. After transferring the mash to the tun, the coarse part of the suspended particles, mainly the husks, reaches the bottom of the tun first. The smaller Oberteig particles sediment slower and are therefore separated afterwards [8]. Newton's second law in combination with the Stokes' equation (Eq. 1) is a simple method

used to estimate the terminal sedimentation velocity of single, spherical particles in laminar regime [9].

$$v = \frac{d^2 \cdot g \cdot \Delta\rho}{18 \cdot \eta} \quad (\text{Eq. 1})$$

This constant 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 . It becomes clear that it is possible to substantially influence the sedimentation behavior of the particle when changing its size. Hence, a sufficient flocculation of the fine particles could affect their velocity so they sediment similarly to the coarser ones. Eventually, this may lead to a more homogeneous filter cake (with no or less top layer), which should accelerate the whole process.

After the preliminary cake formation, the wort begins to flow through the recently formed porous medium [10]. The phenomenon can often be described using Darcy's law (Eq. 2).

$$Q = \frac{\Delta p \cdot A \cdot k}{\eta \cdot h} \quad (\text{Eq. 2})$$

Darcy's law states the proportionality between the volumetric flow rate Q and pressure difference Δp . Q also depends on the cross-sectional area A , permeability k , filtrate viscosity η , and height of the porous medium h . The permeability is characteristic for the given porous structure and it can be estimated using the similarly common equation 3 by *Kozeny* and *Carman* [11].

$$k = C \frac{\varepsilon^3}{(1 - \varepsilon)^2 \cdot S^2} \quad (\text{Eq. 3})$$

The permeability is thus a function of the porosity ε and the specific surface S in the filter cake. At the same time, both variables depend on the particle size. The bigger their diameter, the bigger the void space between them and the smaller their specific surface. As a result, both the permeability and the volumetric flow rate are enhanced, which should also accelerate the wort production.

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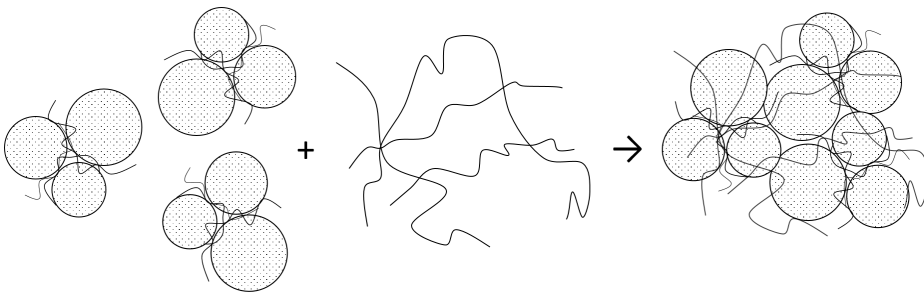


Fig. 1 General effect of clarifying agents on micro-flocs

In summary, an enlargement of the fine particles in the mash through agglomeration should therefore be beneficial, not only during the sedimentation stage in the lauter tun, but also during the subsequent separation stage through the husks. The sedimentation should occur faster and the permeability should be improved.

The agglomeration can be achieved using clarifying agents. When added to a suspension, these substances (often polymers) take advantage of their own structure and even of the polarity of the suspended solids so as to bind them and build flocs of larger size and mass. As a consequence, particles (e.g., colloids) start to settle much more quickly than they would in their absence, making them easier to retain and accelerating clarifying processes [12].

There are several agglomeration mechanisms which depend on different forces [13]. The ones that do not need material bonds, such as Van-der-Waals forces, are of particular interest. Whether these interactions between the polymers and the suspended particles will manifest depends on the nature of both substances. In general, each particle or (if the suspension is not stable enough or a coagulant has been used [14]) small flocs of particles accumulate on the flocculant molecules, forming macro-flocs. A successful agglomeration of micro-flocs using polymers is illustrated in figure 1.

To achieve this, stable macro-flocs have to be formed after adding a specific dosage of the polymer. An insufficient amount will not clarify the liquid fast enough and an excessive amount will contaminate it or even stabilize the suspension, thus preventing flocculation. Therefore, the flocculants' interactions with the suspended particles have to be known. This is commonly studied in water treatment empirically by means of so called jar-tests [15], with which different combinations of clarifying agents at different concentrations are analyzed qualitatively (see experimental method).

After determining the most advantageous flocculant and its corresponding effective concentration, it is necessary to scale up the dosage and put it to the test through the process of interest. Afterwards, it is likely that the dose will require minor adjustments to achieve the expected optimization. For this reason, the experiments of this study were split into two series: the first was to analyze the effects of different flocculants on Oberteig

particles in the jars, and the second was to evaluate their performance in a laboratory lauter tun.

2 Materials and Methods

Pilsner malt (type Laureate, Malteurop GmbH) was used for the experiments. It was conditioned with 2 m% deionized water before being ground twice with a two-roller mill (model 16/16, Künzel Maschinenbau GmbH) with the grinding gaps 1 and 0.4 mm.

The first experiment series was performed only with Oberteig. To recreate it, the malt grist was classified according to the MEBAK procedure [16] and 25 g of both the fractions between 250 and 125 μm and smaller than 125 μm were used as samples. Each 50 g grist sample was mashed in a congress mashing bath with 200 ml deionized water according to the temperature program depicted in figure 2.

The jar-tests were started immediately after finishing the program. The basic experimental equipment is shown in figure 3.

The transparent containers had built-in stirrers to mix the Oberteig suspensions with the flocculants. It was possible to perform various tests simultaneously and to compare them in situ with a

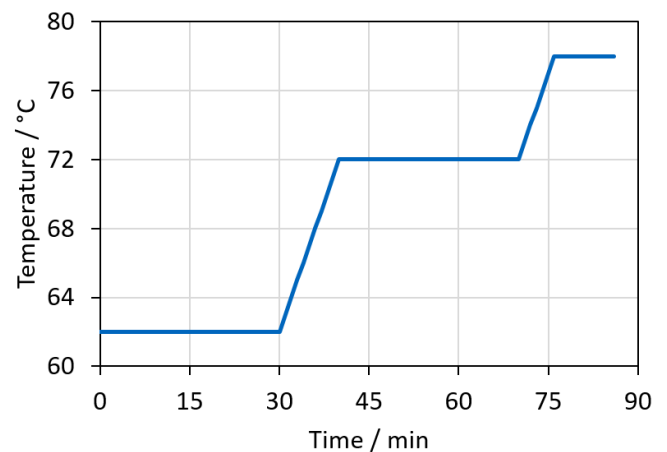


Fig. 2 Temperature program of the mashing process

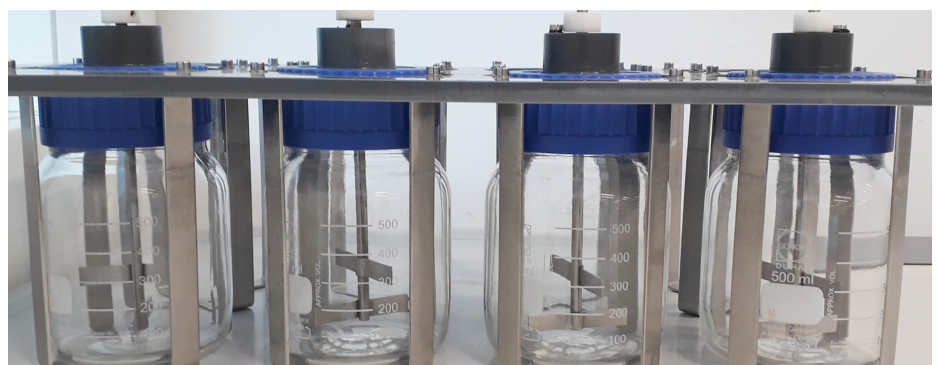


Fig. 3 Experimental equipment used for jar-tests

Table 1 Flocculants used in the jar-tests and concentrations based on Oberteig mass

Flocculant	Manufacturer	Tested concentrations [m%]
Polyvinylpyrrolidone (PVPP)	BASF SE	[5; 10; 15]
Polyvinylpyrrolidone (PVP) K90	Carl Roth GmbH	[1.5; 2; 3]
Polyvinylpyrrolidone (PVP) K30	Carl Roth GmbH	[1.5; 2; 2.5]
Gelatin (type A, 80 % protein)	Gelita AG	[2.5; 3; 4]
Kieselsol (15 %)	VIERKA GmbH & Co	[0.3; 0.45; 0.6]

reference sample. The method was also used to find the effective concentrations of the flocculants.

The samples were transferred from the mashing containers to the equipment in figure 3 and clarifying agents were added, varying type and concentration (Tab. 1). The containers were kept at 78 °C in the mashing bath, while the suspensions were stirred for 10 additional minutes. Afterwards, the setup was taken out of the bath and filmed to perform a qualitative visual analysis of the

clarification according to the sediment height, settling velocity, and turbidity of the clarified liquid.

The K-notations 30 and 90 are proportional to the mean molar mass of the PVP molecules. The results of this experimental series determine whether the flocculants positively affect the suspension and provide a first estimate of the optimal concentration for the following experiments. Next, part of the same samples were analyzed with the Turbiscan (MA 2000, Formulation) and the software Turbisoft (version 1.2.1). The equipment measures infrared light ($\lambda = 850 \text{ nm}$) transmission through the suspension periodically to calculate the peak settling velocities of the fine particles based on the sediment height and, thus, quantify the observed clarifications.

The second experimental series was performed using 500 g of all the grist particle sizes. The malt was mashed with 2 liters of deionized water in a reactor LR-2000.70 (IKA Works, Inc.) according to the program in figure 2. After finishing this step, the adjusted dosage was added and the mash was stirred for 10 minutes. The lautering step was carried out in a laboratory lautertun with a 10 cm diameter. After transferring the mash, it was allowed to settle for 10 minutes before starting the separation. The filtered wort mass was monitored live using an automated precision scale (KB 10000-1N, Kern & Sohn GmbH). At the end of each run, the wort's quality was characterized by measuring its density with a digital density meter (Anton Paar GmbH). All the experiments were performed threefold to ensure their reproducibility.

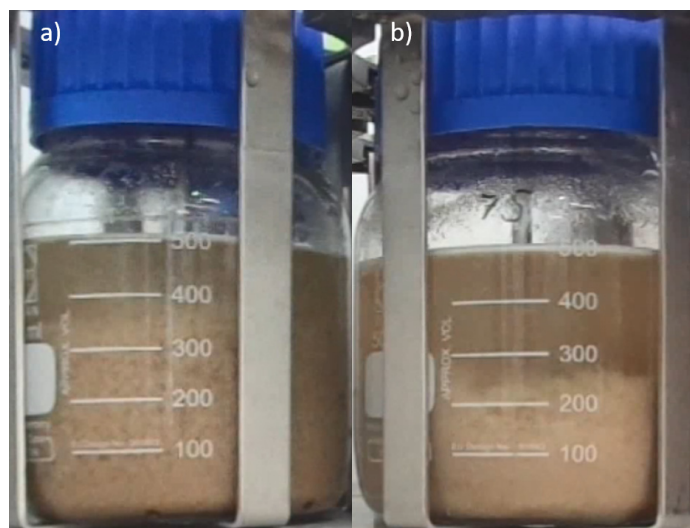


Fig. 4 Jar-tests after 1 min settling time with a) Reference, b) PVP K30

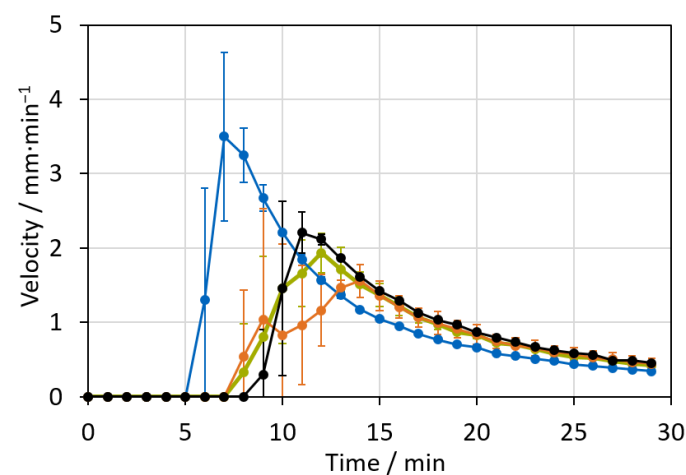


Fig. 5 Settling velocities of particles treated with PVPP. Reference (black), 5 m% (green), 10 m% (dark blue), 15 m% (orange)

3 Results and Discussion

The jar-test results were qualitative comparisons of the flocculant effects with an untreated reference sample based on floc formation, sedimentation velocity, and liquid clarification. It was first observed that the selected clarifying agents did have an influence on the sedimentation of the suspended particles. As a consequence, further tests were done to estimate the best possible flocculant concentration per sample.

Figure 4 is an example of the jar-test results, where the reference and a sample treated with 75 mg PVP K30 are compared. Both images were taken one minute after starting the test and it is already possible to notice the differences. The treated particles exhibit a greater sedimentation velocity because the sediment occupies a smaller volume after the same period. This also means that a larger liquid volume was clarified. It was possible to observe flocs at the surface of the sediment as well. Cases like this qualified for

Table 2 Summary of peak settling velocities and respective effective dosages

Flocculant	Most effective dosage [m‰]	Peak settling velocity [mm·min ⁻¹]	Time of peak velocity [min]
–	–	2.21	11
PVPP	10	3.50	7
PVP K90	2	2.24	5
PVP K30	2	2.78	7
Gelatin	3	2.46	9
Kieselsool	0.45	1.98	5

Table 3 Mean relative time savings after adjusted flocculant dosage based on Oberteig mass

Flocculant	Most effective dosage [m‰]	Lautering duration at 1,400 g wort [s]	Relative saved time [%]
–	–	4,052	0
PVPP	8.5	1,759	56.6
PVP K90	0.71	1,786	55.9
PVP K30	0.71	1,971	51.4
Gelatin	0.21	1,450	64.2
Kieselsool	0.16	1,650	59.3

analysis in the Turbiscan to further evaluate the settling behavior of the treated particles. An example of the results obtained with this method is shown in figure 5.

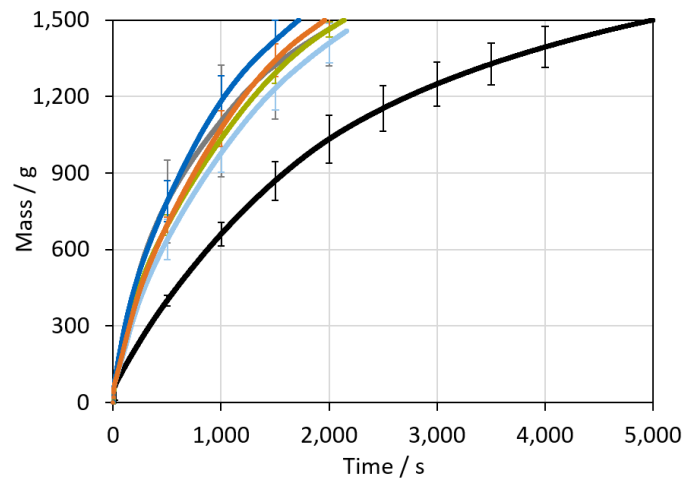
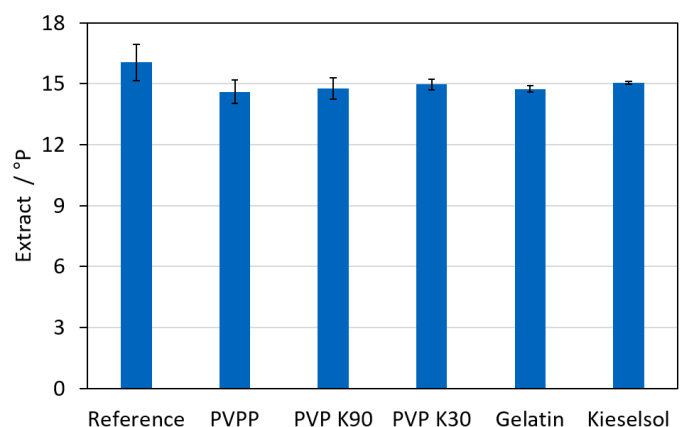
It is clear that the PVPP dosage of 10 m‰ was the most effective for the treatment of Oberteig particles. The blue curve exhibits a much higher peak than the reference (black), while both the previous and the next concentrations could not enhance the settling velocity. The error bars represent the standard deviations of the measurements.

It is also noteworthy how the 10 m‰ dosage destabilized the suspension so that the settling started earlier than in all other cases. Table 2 presents all determined peak settling velocities and times at which they occurred.

Advancing the starting point of the sedimentation was a general result for all used flocculants, which can have a positive effect during the formation of the spent grain cake in the lauter tun. Not all velocities were increased when compared to the reference, although some notable changes were measured. Since these experiments were performed only with Oberteig, it is also necessary to study the effects on a standard mash.

The best results of the first series were scaled up based on the amount of Oberteig present in the standard mash, adjusted to fit the complete particle size spectrum (Tab. 3), and utilized as mash flocculants in the lauter tun. The recorded wort mass curves are shown in figure 6.

The black curve represents the mean values of the reference experiments without treatment. It becomes evident that the effective dosage of all flocculants accelerated the lautering. The colored curves exhibit much steeper slopes and confirm the proposed

**Fig. 6** Wort mass over time (see dosages in table 3). Reference (black), PVP K30 (light blue), PVP K90 (green), PVPP (gray), Kieselsool (orange), gelatin (dark blue)**Fig. 7** Malt extracts present in the different worts

hypotheses. Furthermore, noticeable reductions of the lautering durations were achieved. The results based on an accumulated wort mass of 1,400 g are reported in table 3.

The optimal dosages do not coincide with the estimations obtained from the jar-test. This is because these treated suspensions contain additional species and their behavior varies from the ones containing only Oberteig. For this reason, no less than three different flocculant dosages were studied in the lauter tun to adjust the effective concentrations.

At least a 50 % process time reduction was achieved with every flocculant. The chain length of the polymers appears to correlate to the degree of flocculation of these particles, as shown by the increased savings achieved by K30, K90, and PVPP. These time differences were, however, not statistically relevant. The fact that the PVP dosages had effects comparable to that of the PVPP dosage, in spite of the much smaller amounts, is worthy of remark. This could ultimately have important financial consequences. The most noticeable effects were observed after adding Kieselsol and gelatin, which can be explained by considering the way they interact with the suspended particles. While the former was achieved using a very small dosage, the latter accelerated the separation the most.

The quality of the wort was monitored to detect changes in the malt extract yield caused by this process strategy. The wort density was measured after every experiment and the results are reported in Plato degrees in figure 7.

The extract concentrations in the reference worts were higher on average. The addition of flocculants reduced the mean relative extract yield by not more than 10 %. This was expected, given that bigger particles make washing processes more difficult. Mass transfer phenomena are accelerated by larger contact surfaces and, hence, smaller particles. Nonetheless, using 95 % confidence intervals as error bars, some overlap can be observed. The most concentrated treated worts are similar to those in the lower portion of the references.

4 Conclusion

The effects of diverse flocculants on the fine Oberteig particles were studied by means of a water-treatment method. The effective concentration of each polymer was determined systematically in order to agglomerate the particles and form stable flocs. Thus, their sedimentation behavior was changed and the process was notably accelerated after building a more homogeneous and more porous spent grain filter cake. The dosage of every flocculant achieved to halve the duration of the separation process. The effects caused by PVP, Kieselsol, and gelatin were remarkable, which is why their interactions with the suspension should be further analyzed. Moreover, experimental series with focus on washing the spent grain cakes have yet to be performed. The results obtained thus

far support the hypothesis that agglomerating the fine particles in the mash before lautering considerably increases the wort flow.

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