

# Particle Size Distribution in Wort During Large Scale Brewhouse Operations

F. Kühbeck, W. Back, and M. Krottenthaler

Lehrstuhl für Technologie der Brauerei I, Technische Universität München, Weihenstephaner Steig 20,  
D-85354 Freising, Germany

T. Kurz

Institut für Lebensmitteltechnologie und Lebensmittelchemie, Fachgebiet Lebensmittelverfahrenstechnik,  
Technische Universität Berlin, Amrummer Str. 32, D-13353 Berlin, Germany

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*This investigation highlights the application of particle size distribution measurement in the brewing process steps of mash separation, wort boiling, and wort clarification. Large-scale trials carried out in seven industrial operations applying different process techniques resulted in the appearance of fine (5  $\mu\text{m}$ ), medium (20–25  $\mu\text{m}$ ), and coarse particles (>200  $\mu\text{m}$ ). During mash separation, the medium and coarse particles were removed while fine particles became dominant and were responsible for the remaining lauter turbidity. Further, prior to start of wort boiling medium particles representing hot trub are formed while the relative portion of fine particles is reduced. This formation is substantially completed when wort boiling temperature is reached, a fact not conforming to current expectations. The coarse particles occurring during wort boiling were identified as originating from hop pellets, while the addition of soluble hop extracts did not cause this signal. Finally, wort clarification by whirlpooling, which is a hydrodynamic separation step, could be described by reduction of coarse and partly of medium particles, while fine particles dominate the resulting wort. The particulate changes observed seem to correlate with chemical analytes conventionally used to characterize these process steps. © 2007 American Institute of Chemical Engineers AIChE J, 53: 1373–1388, 2007*

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## Introduction

In contrast to turbidity measurements, which provide only an indication of the overall content of a sample, particle size distributions increase analytical information by providing additional structural information. A comparison between a particle size and a turbidity measurement was presented by Annemüller et al.<sup>1</sup> While the measurement of turbidity detects the properties of the substances causing turbidity in toto, particle size

measurement detects single particles in terms of their number and size. Therefore, the selectivity of particle sizing is higher compared with turbidity measurement. When dealing with low particle concentrations, particle sizing shows higher accuracy vs. turbidity measurement, however, only turbidity measurement allows calibration and measurement for higher solid contents [ $>150,000$  particles ( $>1 \mu\text{m}$ )/mL]. Particle sizing is independent of the color of the medium, whereas turbidity measurement is not. While particles of  $<1 \mu\text{m}$  are difficult to detect, turbidity measurement also detects colloidal turbidity.<sup>1</sup> Overall, particle size measurement may allow further information to be gained to describe and improve the process engineering, e.g., applied in the brewing industry.

Correspondence concerning this article should be addressed to F. Kühbeck at Florian.Kuehbeck@wzw.tum.de.

While the measurement of turbidity is widespread in the field of brewing, the measurement of particle size distribution is used only rarely. This was due to the lack of affordable equipment in the past. The state-of-the-art equipment available today is highly sophisticated, allowing fully automated data processing, and therefore provides an easy to handle and reliable method. However, for technical reasons, such as heat sensitivity and need for precise alignment of the optical components, it is still a wish to apply the system online in practical brewing operations.

Regarding the measurement and technological relevance of turbidity or particle size distributions in the field of brewing the following applications are known: grist fineness,<sup>2–5</sup> mash,<sup>2,3,6–9</sup> wort,<sup>10–13</sup> spent grain press liquor,<sup>14</sup> hot trub,<sup>15–21</sup> cold trub,<sup>22–28</sup> beer filtration properties,<sup>29–31</sup> and final beers.<sup>13,32–34</sup> Additionally, properties of the raw materials and their influence on the brewing process were investigated.<sup>35–37</sup> Wort clarification operations were described by particle or turbidity measurements for hot wort (whirlpooling)<sup>38–42</sup> and cold wort (cold trub separation).<sup>23,24,43</sup> Particles occurring during fermentation and their properties were also the focus of research.<sup>44–49</sup> In terms of the final beers, the nonbiological stability of filtered beer,<sup>50–52</sup> chill haze,<sup>53–55</sup> and the stability of yeast containing wheat beers<sup>37,56–58</sup> were of particular interest. Beside the intermediate and final products of beer processing, such as wort and beer, particle size distribution measurements were applied to characterize process aids such as kieselguhrs, poly(vinylpyrrolidone), and others.<sup>1,13,59–63</sup> Although particle size distribution measurements were used for research purposes, they never found much favor in industrial beer production applications as a routine method for reasons mentioned earlier.

In more detail, Lotz et al. undertook particle size distributions of ground malt and found particle sizes of <5 and ~20  $\mu\text{m}$  as being dominant.<sup>5</sup> Stewart and Martin investigated the particle size distribution of a cloudy all-malt wort produced in a pilot brewery, which was not boiled and no hops were added. They found a wide volume distribution from <0.1 to 100  $\mu\text{m}$  in diameter, with the largest percentage of particles being around 7  $\mu\text{m}$ .<sup>13</sup> For spent grain press liquor, particle sizes range from 0.25 to 100  $\mu\text{m}$  with more than 50% thereof being smaller than 8  $\mu\text{m}$ . The origin of these particles is seen in fragments of proteinaceous membranes.<sup>14</sup> According to van Haecht et al. the volume of sedimentable matters in wort after boiling depends on the intensity of turbid wort pumping during mash separation and on the boiling time.<sup>41</sup> In terms of the particle size of hot trub a range of 30–80  $\mu\text{m}$  is reported, which was published already in 1937 by Enders and Spiegl<sup>15,16</sup> and was confirmed by others.<sup>17,18</sup> Ghosh and Sommer investigated into cold trub and its flotation and found median values from 8 to 9  $\mu\text{m}$ .<sup>23,43</sup>

As a result, investigations regarding the particle size distribution of selected samples throughout the brewing process were presented in the past. However, in most of the cases detailed information in terms of the respective brewing process (e.g. mash separation method, wort turbidity, boiling parameters, wort clarification) are missing. Further, there is a lack of information regarding the particle size distribution in wort along the brewhouse procedure, particularly for mash separation, wort boiling, and wort clarification as subsequent process steps. Additionally, it has to be considered that from the brewing engineering point of view quite a big number of innovative

**Table 1. Technological Key Figures of Wort Boiling for All-Malt Beers<sup>104,105</sup>**

Parameter	Setpoint of Cast Wort	Setpoint of Chilled Wort
N coag., mg/L	15–25	–
TBAN	<45	<60
dTBAN (to kettle-up)	≤15	–
DMS free, $\mu\text{g/L}$	<100	<100

N coag., coagulable high molecular nitrogen compounds; TBAN, thiobarbituric acid number; dTBAN, increase of TBAN value during wort boiling; DMS, dimethyl sulfide.

mash separation and boiling techniques were implemented in the last two decades. These include improvements of the classic lauter tun concept, implementation of new lauter tun, and mash filter concepts as well as innovative control principles.<sup>4,5,9,64–82</sup> Innovations have been even more extensive in the field of wort boiling. Beside the conventional internal and external boiler (tubular heat exchanger) novel boiling technologies such as thin-film evaporation, dynamic low pressure boiling, wort stripping, and vacuum evaporation were introduced.<sup>78,83–103</sup> Quite a number of basic investigations were undertaken to characterize and optimize the main technological parameters of boiling such as evaporation rate, dimethyl sulfite (DMS) content, thiobarbituric acid number (TBAN), and coagulable nitrogen compounds (N coag.) (Table 1<sup>104,105</sup>). However, beyond this important work there is a lack of knowledge in enhanced characterization of the applied process techniques. More to the point, particle size distribution changes during mash separation when switching from turbid to clear wort draw-off have not yet been adequately described. How do the different applied techniques, i.e. a lauter tun or a mash filter, influence the particle sizes in wort? How do boiling process technologies, which represent either “more gentle” or “vigorous” boiling philosophies, differ in terms of particle size distribution and chemical properties of the resulting wort in this matter? Or more specifically: Do modern gentle boiling techniques with low maximum temperatures of boiling provide a different particle size distribution compared with conventional boiling procedures with higher heating medium temperatures?

The aim of this investigation is, therefore, to gain systematic knowledge about particle size distributions of wort in different stages of the brewhouse procedure in the process steps of mash separation, wort boiling, and wort clarification. Besides basic information about particle sizes during mash separation and boiling, a variety of different industrial-scale mash separation and boiling systems (Tables 2 and 3) were investigated to provide a comparison of particle size distributions between conventional and novel process techniques and correlate those with conventional analytical parameters, such as N coag. and TBAN.

## Experimental

### Particle size distribution

For the measurement of particle size distribution in the range of 0.1–875  $\mu\text{m}$  a laser diffraction sensor (HELOS/BF, Sympatec, Clausthal-Zellerfeld/Germany) equipped with a HeNe laser ( $\lambda = 632.8 \text{ nm}$ ;  $P = 5 \text{ mW}$ ) and a wet dispersion unit (SUCCELL, Sympatec, Clausthal-Zellerfeld/Germany) was

**Table 2. Technical Data of Mash Separation and Separation Systems of Industrial Brewing Operations (A–G) and Pilot Plant (H) Investigated**

Brewery	A	B	C	D	E	F	G	H
Lauter system	Lauter tun	Lauter tun	Lauter tun	Lauter tun with clearance	Lauter tun	Lauter tun	Mash filter	Lauter tun (pilot)
Year of manufacturing	1980	1988	1967	2002	1990	1981	2004	1989
Inner diameter, mm	4800	9000	4040	4000; clear.: 2000	9200	5400	–	340
Total volume, hL	217.2	1272	189.7	110	1328	455	–	0.6
Specific load, kg/m <sup>2</sup>	210.5	179.2	136.5	183.4	198.6	218.3	31.7	33.06
Slot width of false bottom, mm	0.7	0.7	1.5	n.a.	0.7	0.75	–	0.7
Number of knives, m <sup>-2</sup>	1.33	n.a.	1.56	1.48	1.58	1.22	–	11.02
lauter time (first + weak wort), min	128	150	102	106	137	129	80	120–130
Total load, kg	3810	11,400	1750	1740	13,200	5000	5800	12
Effective lautering area, m <sup>2</sup>	18.10	63.62	12.80	9.49	66.48	22.90	183	0.363
Volume of lauter wort, hL	192	676	110	118	750	315	340	62

Systems vary in terms of age (manufacturing year) and process engineering (lauter tun = horizontal bed filtration supported by milled false bottom; mash filter = vertical filter bed compartments supported by PP glove); min, minute; hL, 100 L; n.a., not available.

used. Zeroing was carried out with deionized water prior to each run. For sample measurement wort of ~20°C was added to the water until the optical density of the resulting suspension was in an optimum range (10–15%). Prior to and during measurement dispersion was achieved by stirring (stirrer setpoint: 50%) the content of the sample basin (approximate volume: 500 mL) and pumping its content through a flow cell measuring cuvette (pump setpoint: 80%). Stirrer and pump performances did not exceed the above setpoints in order to avoid foaming of the analyte suspension causing interference. The stainless steel flow cell had a path length of 2 mm. All samples were measured without and with ultrasonic treatment ( $P = 60$  W) in steps (0, 30, 60, 90, 120 s) to provide an optimum dispersion.

### Mash separation and wort boiling systems

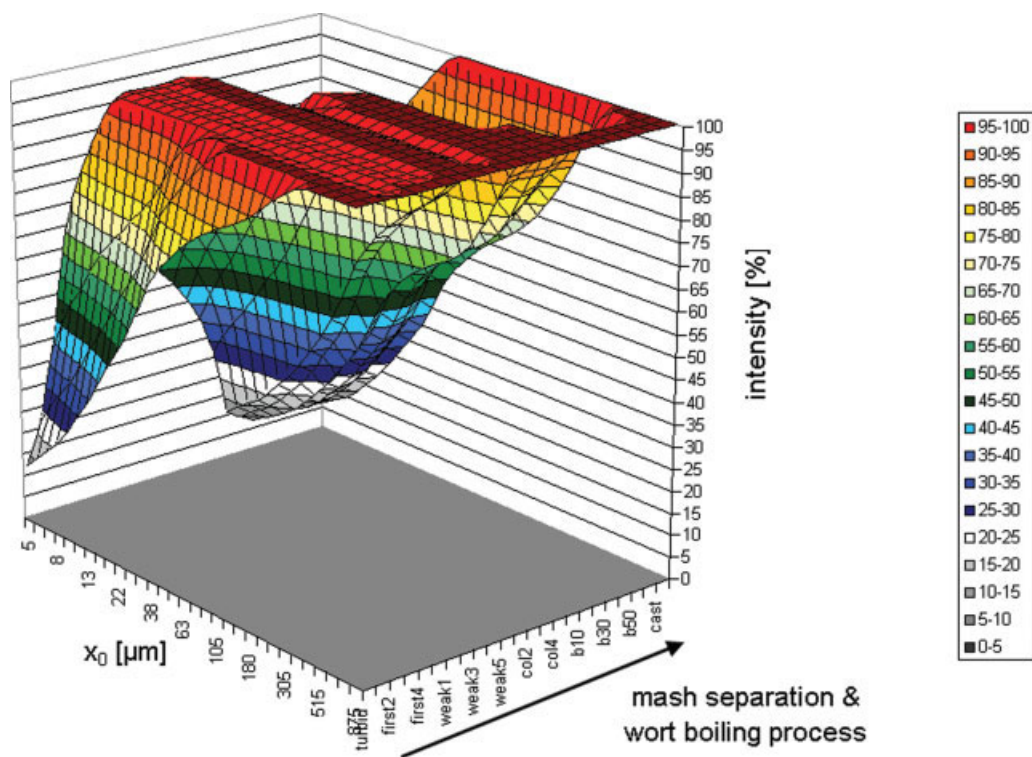
A variety of lauter tuns (filter bed formation by gravity; bed support: stainless steel false-bottom) of different designs and/or from different manufacturing dates and a modern mashfilter

(forced filter bed formation; bed support: polypropylene glove) were investigated. Samples were taken during turbid wort pumping, first-wort draw-off, sparging draw-off, and partly during last-running draw-off. In terms of wort boiling seven different systems covering conventional boiling technologies (internal/external tubular heat exchanger) as well as the latest technologies (internal tubular heat exchanger with forced circulation flow, thin film evaporation, wort stripping, vacuum evaporation) were investigated. Additionally, pilot scale trials were undertaken applying a method and system for achieving clear lautering without raking and conventional hot trub separation by whirlpool as described.<sup>106</sup> Within the pilot scale brews a variation of hopping was investigated with no hops, and adding CO<sub>2</sub> extract hop (Hopsteiner, Mainburg/Germany) and pellet hops (type 45; Hopsteiner, Mainburg/Germany) to boiling wort. Samples were taken during wort collecting and heating-up of kettle wort, of kettle-up wort (start of boiling), of boiling wort in intervals of 10 min, of cast wort and hot clarified wort when half of the wort had been chilled. Details of all systems are presented in Tables 2 and 3.

**Table 3. Technical Data of Thermal Treatment, Boiling Systems, and Wort Clarification of Industrial Brewing Operations (A–G) and Pilot Plant (H) Investigated**

Parameter	A	B	C	D	E	F	G	H
Boiling system	Gentle boiling + wort stripping	External	External + wort stripping	Thin film evaporation	Internal	Internal with forced feed	External	Wall heating (pilot)
Year of manufacturing	1970/2005	1981	1990/2000	1998	1992	2006	1998/2005	1989
Inner diameter, mm	–	5800	4800	3000	5982	4000	4200	420
Boiling temperature at boiler outlet, °C	100	103	105	98.5	101–102	100	102.5	98–99
Boiling temperature in kettle, °C	98 (60 min) + 99.5 (5 min)	102–103	98	96–97	98.5	99	99.7	100
Boiling time, min	60 + 5	55	35	47	65	70	50	60–90
Evaporation rate, %/h	1 (boil.)/60 (evapor.)	7.6–8.7	13.7	5.7	5.5–6.5	3	6	10–12
Volume of cast wort (chilled), hL	230	620	102	108	750	300	390	0.6
Heating area, m <sup>2</sup>	n.a.	396	19.7	n.a.	78.7	33.8	300	0.14
Whirlpool stand, min	30	15	10	20	25	45	35	15
Wort cooling, min	60	60	45	60	60	60	60	10

Systems vary in terms of age (manufacturing year) and process engineering (gentle boiling = gentle heating of wort and keeping wort <100°C; wort stripping = evaporation step in reduced pressure or vacuum; external = tubular heat exchanger with forced circulation flow; internal = tubular heat exchanger with natural or forced circulation flow); min, minute; hL, 100 L; h, hour; n.a., not available.



**Figure 1.** Exemplary particle size sum distribution Q3 of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (F in Table 2); boiling system: internal boiler with forced circulation flow (F in Table 3)]; x axis: equivalent diameter  $x_0$  [ $\mu\text{m}$ ]; y axis: process step [turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool)]; chilled = chilled wort (after whirlpool)]; z axis: peak intensity Q3 [%].

[Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

### Sampling of wort and analyses

Wort samples of  $\sim 0.4$  L (pilot scale trials) or 1.5 L (industrial scale trials) were taken from the wort pipeline (lautering sample) or directly from wort kettle (collecting/kettle-up/boiling) in intervals of 10–15 min depending on the process step, sealed, and were immediately cooled to  $\sim 15^\circ\text{C}$  by water bath. As one aim of this investigation is to gain information about the different stages of mash separation and wort boiling, cooling of samples right after sampling is necessary to stop thermal reactions, which would otherwise continue to take place in a hot sample. On the other hand, cooling of samples to  $0^\circ\text{C}$  or even deep freezing might have an influence on the particle composition of the sample content, and therefore, on the particle size distribution. Permanent and nonpermanent hazes occurring due to cooling to  $0^\circ\text{C}$  are well known in the field of brewing. As a consequence, the samples for distribution measurements were kept at room temperature until analyses. By taking the samples hot and immediate sealing prior to cooling microbiological stability was achieved. All distribution measurements were done at room temperature within 12–18 h after sampling and after homogenization of samples by manual shaking. For all other analyses further batches of samples were stored at  $0^\circ\text{C}$  until analysis and manually homogenized right before analyses.

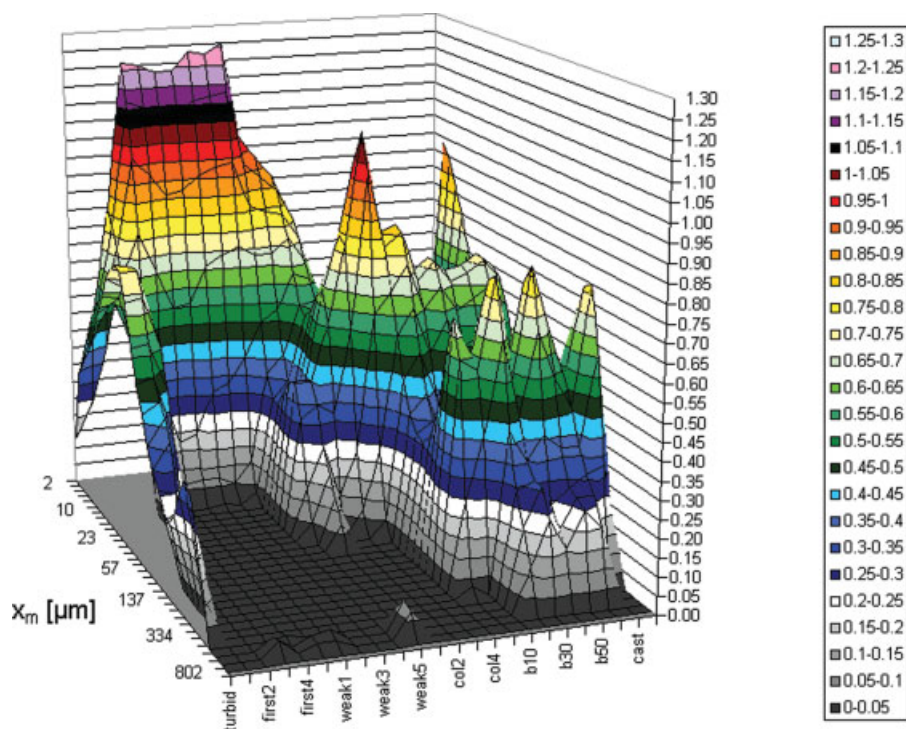
pH, extract, color, N coag., and TBAN analyses were carried out according to standardized procedures of MEBAK II 2.14, 2.10.2.3, 2.13.2, 2.4, and 2.8.2, respectively.<sup>104</sup>

### Elimination of peaks by calculation

For purposes described later, single peaks of distribution diagrams were eliminated by calculation. This was achieved by graphically determining the size of the respective peak to be eliminated in the sum distribution and arithmetic recalculating the peak size of the remaining peaks in that way that their areas sum to 100%. For example, the diagram consists of three peaks A, B, and C, of which C is to be eliminated. The peak areas are like 50% (A), 20% (B), and 30% (C). The recalculated values are:  $A_r = 50\% \times 100/(100-30) = 71.4\%$ ;  $B_r = 20\% \times 100/(100-30) = 28.6\%$ . The recalculated ratio of  $A_r:B_r$  is  $71.4\%/28.6\% = 2.5$  as was prior to calculation ( $A:B = 50\%/20\% = 2.5$ ).

### Presentation of particle size distributions

Particle size distributions may be displayed as sum distribution (Figure 1) as well as density distribution (Figure 2). Since in the case of this investigation density distributions seem to make it easier to detect differences between distributions, the following results are presented in the density distribution in semilogarithmic diagrams. A typical presentation of particle size distribution of mash separation (separating liquid and solid phase of mash into wort and spent grains) and wort boiling process (most intensive thermal process step of brewing) contains the equivalent particle size  $x_m$  as  $x$  axis, the process step as  $y$



**Figure 2.** Exemplary particle size density distribution  $q_3$  of wort during mash separation, wort boiling, and wort clarification (mash separation system: lauter tun (F); boiling system: internal boiler with forced circulation flow (F));  $x$  axis: equivalent diameter  $x_m$  [ $\mu\text{m}$ ];  $y$  axis: process step [turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)];  $z$  axis: relative peak intensity  $q_3$  [ $1/\mu\text{m}$ ].

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axis and the peak height as  $z$  axis (Figure 2). To simplify the peak identification a top view maybe suitable (Figure 3). Herein the particle size is displayed as  $x$  axis, the process step as  $y$  axis and the color within the diagram represents the peak height (equivalent to  $z$  axis).

## Results and Discussion

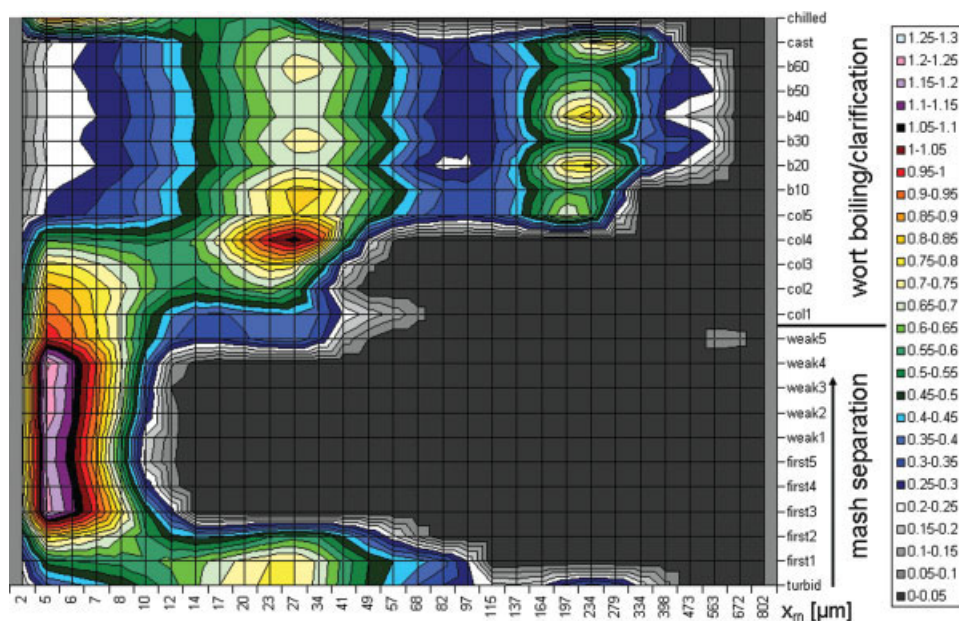
From the particle size top view (Figure 3) it is understood that essentially three different peaks occur during mash separation and wort boiling: at  $\sim 5 \mu\text{m}$  (referred as fine particles in the following), at  $\sim 23 \mu\text{m}$  (referred as medium or middle-size particles), and at  $\sim 235 \mu\text{m}$  (referred as coarse particles). This finding confirms the results of Lotz et al. discussed earlier.<sup>5</sup> The reason for the occurrence of three distinct groups of peaks (fine, medium, and coarse) in all investigated breweries could be either due to the similar raw materials or to the milling systems. Indeed, when applying different milling systems, such as roller mills for lauter tuns or hammer mills for mash filters, substantially the same classification of particles occurred (see later). Therefore, the milling procedure seems to be of minor importance in this case. On the other hand, all breweries within these trials applied malted barley to produce a lager type beer. Since the raw material barley was genetically similar for all trials, the respective malts also had similar characteristics. It is well known that barley malt kernels contain smaller and larger particles of which the smaller have a diameter of

around  $5 \mu\text{m}$  and mainly contain protein or fractions of proteinaceous membranes.<sup>14</sup> In contrast, the larger ones have a diameter of around  $20 \mu\text{m}$ , with starch being their main component. As discussed earlier, most of the authors found peaks at  $7\text{--}8 \mu\text{m}$  in cloudy unhopped wort or spent grain press liquor,<sup>13,14</sup> which basically confirms the presence of  $5\text{-}\mu\text{m}$  fraction found here.

Since a presentation of Figure 3 contains  $\sim 750$  data points, it seems to be suitable to simplify the presentation by mainly focusing on only the three peaks mentioned earlier when discussing particle size distributions throughout the process steps of mash separation, wort boiling, and wort clarification, since these peaks were characteristic for all industrial processes investigated.

### Mash separation

After completing the mashing process and transfer of mash to the lauter tun or mash filter the mash separation step starts by turbid wort pumping, to form a filter layer and to lower the turbidity of the resulting wort for technological reasons. In the recycling wort particles of medium size ( $\sim 23 \mu\text{m}$ ) were dominant, while fine and coarse particles ( $5$  and  $235 \mu\text{m}$ ) were of minor importance (Figures 3, 4, 6, 8). After turbid wort pumping the draw-off of first wort ("first" in figures) starts. While drawing-off the first wort the coarse particles disappeared ("first1"), later followed by the medium particles ("first3"),



**Figure 3.** Top view of an exemplary particle size density distribution  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (F); boiling system: internal boiler with forced circulation flow (F)]; x axis: equivalent diameter  $x_m$  [ $\mu\text{m}$ ]; y axis: process step [down: mash separation; top: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; z axis: relative peak intensity  $q_3$  [ $1/\mu\text{m}$ ] expressed as colors; considerable signals at 5, 23, and 234  $\mu\text{m}$ .

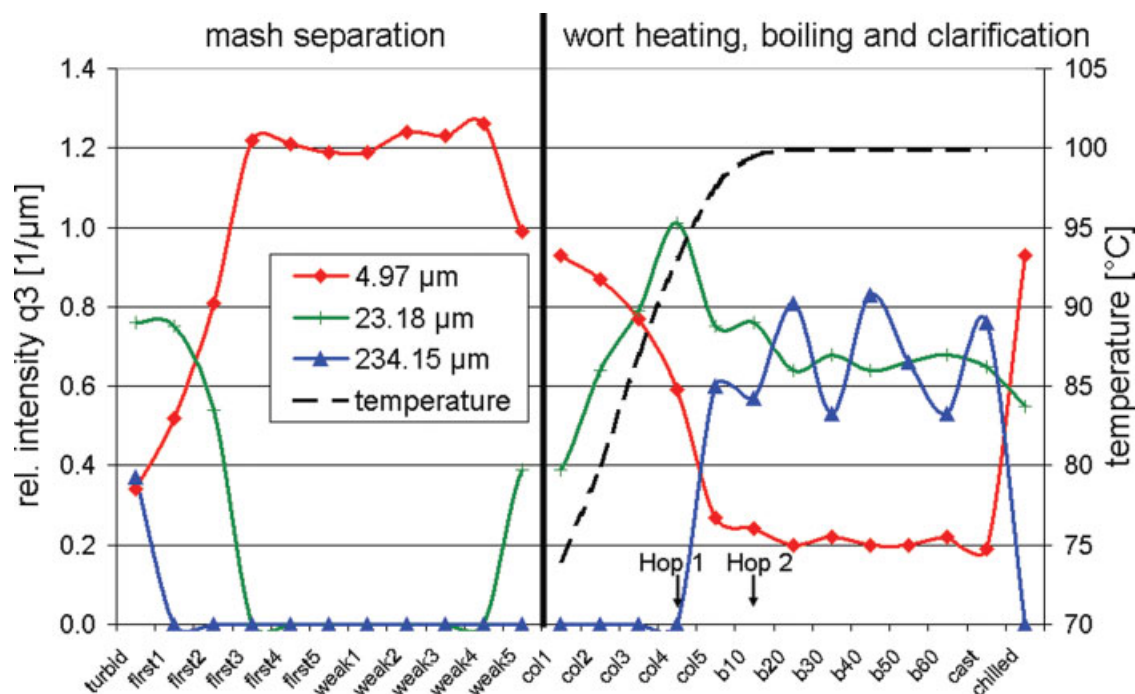
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with the fine particles became dominant. When first wort draw-off is completed, sparging follows when hot water is sprayed on top of the filter cake to wash out the remaining extract. As the water penetrates through the filter layer the extract content of the draw-off starts to decrease successively. This so-called weak wort draw-off continues until the end of mash separation and is stopped when the extract content of the weak wort reaches a certain lower limit ( $\sim 1^\circ\text{Plato}$  ( $=^\circ\text{P}$ )). Sparging can be done continuously or batch-wise, with the number of water additions depending on the individual brewhouse procedure and may be in the range of 1–3. At the beginning of weak wort draw-off fine particles dominated, whereas middle and coarse particles did not occur. At the end of lautering the medium particles reappeared due to raking, that is, vertical cutting of the filter layer by applying a number of knives in slow motion. In some cases, when deep raking close to the false-bottom (filter layer support) was applied to increase penetration rate of water through the filter layer peaks of medium particles appeared during draw-off (Figures 4 and 5). This may particularly occur with older lauter tuns or improper raking conditions.

The particle size distributions as discussed here correlate to the draw-off turbidity measured in-line in EBC units. The turbidity decreased rapidly and significantly during turbid wort pumping and reached the lowest value during first wort draw-off, when coarse and medium particles disappeared and only fine particles remained in the drawn-off wort (Figures 5, 7). Throughout all observed lauter systems, the fine particles predominate in the first wort without exception, however, their absolute concentration may vary due to grist composition,

design, and age of the lauter system. Thus it may be concluded that the remaining turbidity of first wort was basically only caused by particles of a size of  $\sim 5 \mu\text{m}$ , while the medium and coarse particles are retained very well by the filter bed of a proper working lauter tun. Therefore, a further reduction of wort turbidity can only be achieved if the retention of this fine fraction is further improved, which should be considered in future developments regarding the design of a lauter tun. Toward the end of lautering, when the medium particles occurred again the turbidity also increased (Figure 5). With older lauter tuns even coarse particles may occur at the end of the process or in rare cases throughout the whole process (not shown). This might be due to an improper process control with unnecessary deep cuts or an unsuitable design of the raking machine. Therefore the adjustment of the process control and the application of a state-of-the-art raking machine are crucial points when trying to lower wort turbidity and particle flow.

Leaving lauter tuns aside for the moment, a modern mash filter was also under investigation. Mash filters are characterized by vertical filter compartments having polypropylene gloves as support instead of a horizontal false bottom (lauter tun). Here basically the same particle sizes occurred as in lauter tuns (Figure 6). From Figure 6 it can be seen that the coarse particles occurred only during turbid wort pumping and here to a lower extend compared with the lauter tuns. The medium particles predominated during turbid wort pumping and first wort drawn and did not decrease before weak wort draw-off, when fine particles became the major fraction. In contrast to a modern lauter tun, the medium particles did not completely disap-



**Figure 4.** Sectional trends of particle size density distributions  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (F); boiling system: internal boiler with forced circulation flow (F)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; temperature during wort collecting and boiling (broken line); x axis: process step [left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: temperature during wort boiling [ $^{\circ}\text{C}$ ]; arrows indicate points of hop additions (hop 1 and 2: pellet hopping).

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pear throughout the entire mash separation process. Toward the end of draw-off the medium size particles increased similarly to lauter tuns as described earlier. In conclusion, this means filtration characteristics of a mash filter differ from a modern lauter tun with mash filter wort having a considerable amount of medium particles additionally to the fine fraction. Thus, if the filtration performance of a modern mash filter is to be improved in terms of the particle composition of the resulting wort, a further reduction of the fine and particularly of the medium particles should be achieved. Possible ways to do so may be an intensified recycling of turbid wort at the start of filtration or an increase of the height of the filter bed layer. In contrast, a modern and properly controlled lauter tun has a different particle composition with a fast reduction of coarse and medium particles at the beginning of mash separation and a complete absence of these throughout the entire draw-off period (Figures 4, 7).

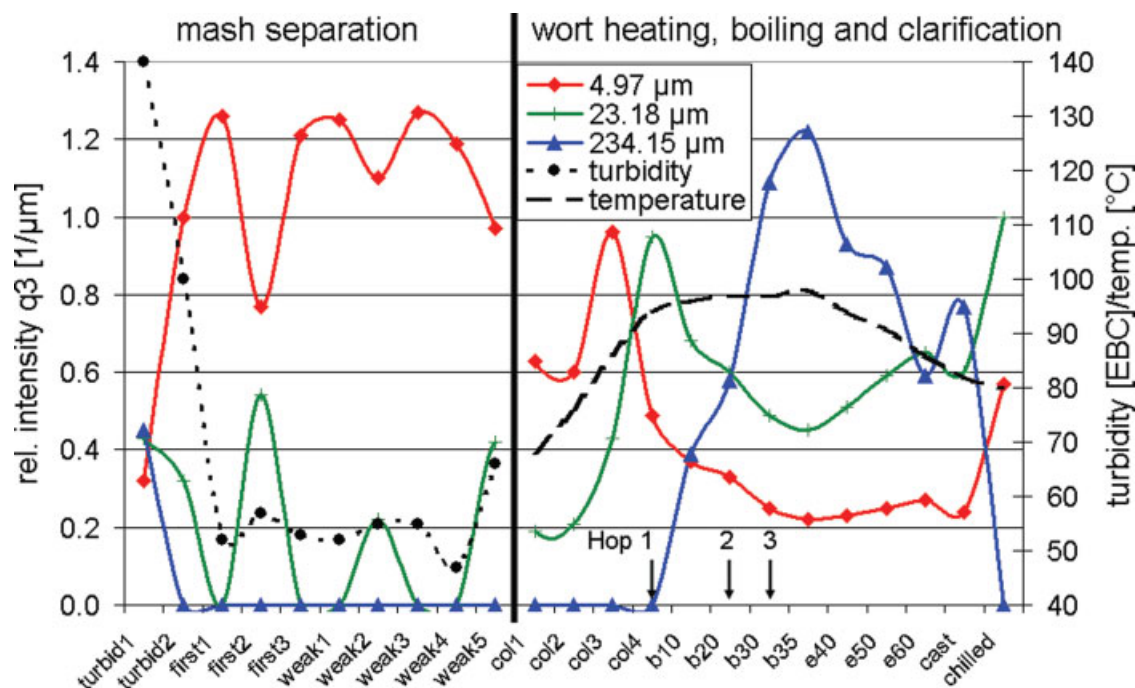
### Wort boiling

The most intensive thermal step within the brewhouse procedure is wort boiling. It is required to sterilize wort, stop enzymatic reactions, evaporate water from wort to an desired extend, remove unwanted wort aroma compounds, improve dissolution and isomerization of hop components, and cause precipitation of wort protein contents to an insoluble coagu-

lum, known as hot break or hot trub. From a technological point of view wort boiling has often been characterized by heat load (expressed as increase in TBAN), evaporation characteristics (evaporation rate), decrease of DMS (evaporation of unwanted flavor compound) and N coag. (formation of hot break).

### Collecting and heating of wort

In modern boiling operations wort is collected in the kettle during mash separation, and heating of wort often starts already when wort reaches a certain level in the kettle before mash separation is completed, to reduce operation time. Because of the heating wort having a temperature of around 90–95 $^{\circ}\text{C}$  when the collection of wort is complete (kettle-up), the time until start of boiling is shortened. Although the reactions taking place during wort boiling are primarily discussed, it is interesting to observe what happens prior to the start of boiling. The wort collected in the kettle prior to heating contained a similar particle size distribution as described for mash separation: fine particles dominate, medium particles were present to a lower extend while coarse particles did not occur at all (Figures 4–7). When heating up to boiling temperature, an increase of the medium particle peak was observed, while the fine particle peak decreased (Figures 4, 5, 7). This means there was a coarsening of particles from the fine to the medium



**Figure 5.** Sectional trends of particle size density distributions  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (C); boiling system: external boiler + wort stripping (C)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; turbidity during mash separation (broken line with points); temperature during wort collecting and boiling (broken line); x axis: process step [left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; en = evaporation, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: turbidity of lauter wort [EBC]/temperature during wort boiling [ $^{\circ}\text{C}$ ]; arrows indicate points of hop additions (hop 1, 2, and 3: pellet hopping).

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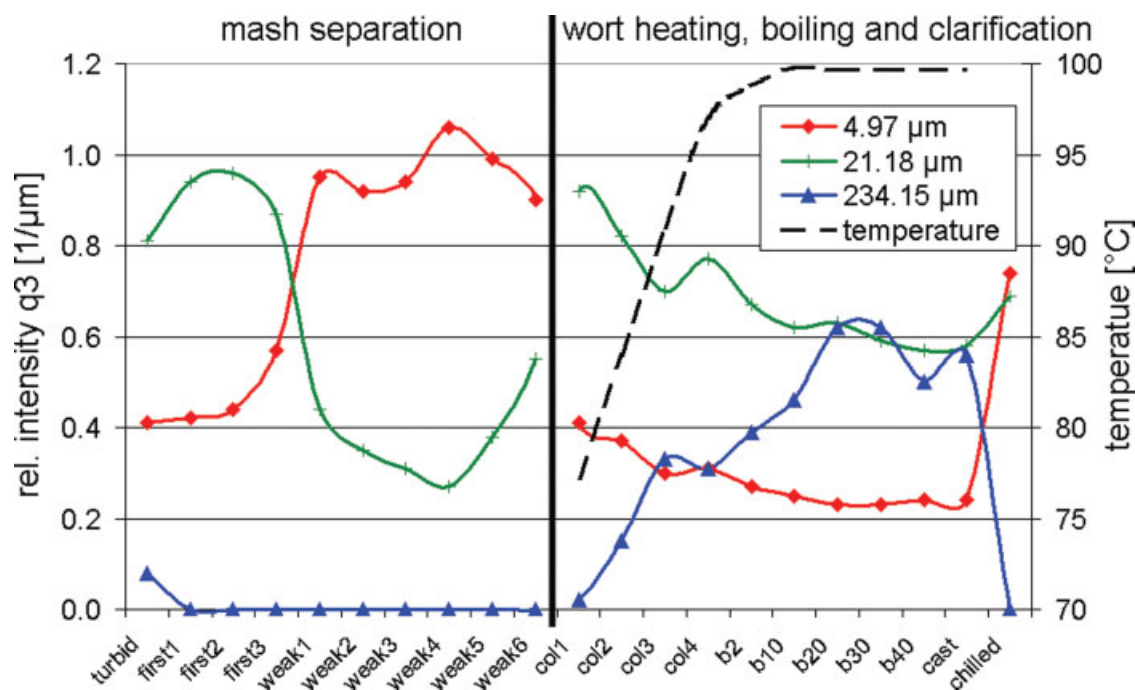
fraction, which is because of the coagulation of mainly protein material accompanied by the adsorption of other substances such as lipids and metal ions. Accordingly, one may conclude the formation of hot break does not only occur during wort boiling as it is mostly described in the literature, but takes already place before the whole batch starts to boil. To confirm this result a pilot-scale wort batch was collected, and heating was started only after mash separation was completed. When homogeneously heating the whole batch (stirring), medium particles occurred only when a temperature of  $98^{\circ}\text{C}$  (boiling temperature) was reached (not shown). Therefore, the formation of hot trub particles requires boiling temperature or a temperature close to it. This seems to be in contrast to the earlier observation that particle formation in large-scale trials occurred right after the start of heating of wort and the portion of medium particles increased while heating. This can be explained since a bypass flow from the wort kettle is heated to boiling temperature in a boiler, which provides sufficient thermal energy to allow the coagulation reaction to take place. Particles are formed in this bypass flow, which is then returned to kettle and mixed with the remaining cooler wort. Since the bypass heating takes place continuously, the amount of medium particles in the kettle continuously increases during wort heating, which was actually observed (Figures 4, 5, 7). This phenomena sup-

ports other observations where a formation of turbidity and/or a strong decrease of free fatty acids were reported prior to wort boiling ( $<100^{\circ}\text{C}$ ), which is due to adsorption to hydrophobic surface areas at hot break formation.<sup>107–109</sup> As the temperature increased not only the signal level at a particle size of 23  $\mu\text{m}$  increased but also the peak extended toward coarser particle sizes (Figure 3). This confirms earlier publications reporting of particle sizes in hot break of 30–80  $\mu\text{m}$ .<sup>15–18,26,110,111</sup> However, one reason for the appearance of medium particles might also be seen in the introduction of medium particles during the late phase of mash separation. Here the observation of a most modern lauter tun gives an important indication: even when no medium particles occurred during mash separation and were therefore not introduced into the kettle during collecting, the medium particle peak increased when wort was heated (Figure 7). In other words, there seems to be a considerable formation of medium particles already during heating. These results were confirmed by pilot-scale trials, where a clear draw-off of wort free of medium or coarse particles was achieved and heating started only after collection was completed (not shown).

#### Boiling phase

At the start of boiling, which was in the range of  $97\text{--}100^{\circ}\text{C}$  depending on the boiling system and the applied pressure a



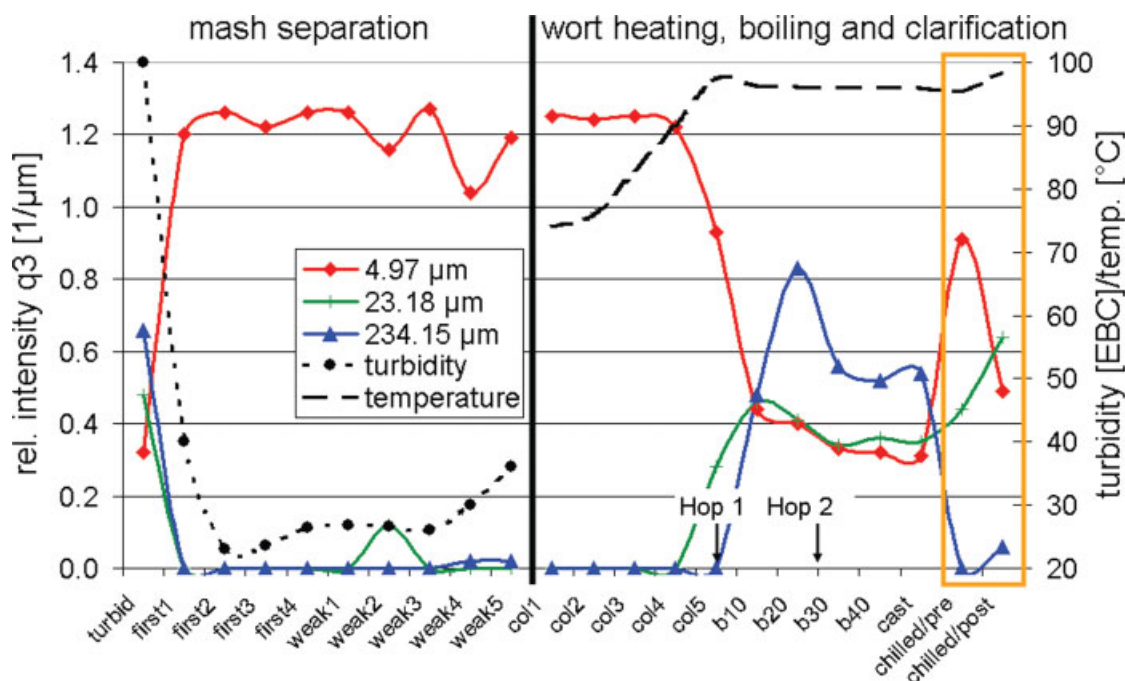


**Figure 6.** Sectional trends of particle size density distributions  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: mash filter (G); boiling system: external boiler (G)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; temperature during wort collecting and boiling (broken line); x axis: process step [left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: temperature during wort boiling [ $^{\circ}\text{C}$ ].

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sudden increase of the coarse particles occurred (Figures 4, 5, 7). This might be caused by hopping when hops were added to the boiling wort mostly in form of dried pressed hop pellets at the start of boiling. This increase did not originate from mash separation since it also occurred even if no coarse particles were introduced during mash separation. By hopping, therefore, coarse particles are introduced into the boiling wort. In practical operations, the hopping dosage is usually divided into two or three additions added at different points of time. In case of Figure 3 the second hopping took place 10 min after start of boiling, after which the coarse particle peak further increased. Alternatively, the first hopping dose can also be provided as hop extract, either as ethanol or supercritical  $\text{CO}_2$  extract, for bitter hopping. In this case the viscous extract is completely dissolved after addition without remaining of any solid particles. This was investigated in one brewery with extract as first hopping dosage and pellets as second hopping dosage. Here the level of coarse particles was zero prior to hopping and remained zero when extract was added to the kettle at the start of boiling (Figure 8). Twenty minutes later the second hopping was performed using pellets and the coarse peak increased to a measurable value (Figure 8). The conclusion, therefore, is that the signals occurring in the range  $>200 \mu\text{m}$  are caused by hop components, particularly originating from hop pellets. This was confirmed by a pilot-scale brew where  $\text{CO}_2$  extract and pellet hoppings were carried out subsequently with a peak at  $>200 \mu\text{m}$  only occurring after pellet hopping (not shown).

At this point it should be noted that the sum of each particle size density distribution as displayed here adds up to 100%. For instance, the signal of a particle class A decreases when a new particle class B is introduced such as during hopping with pellets, despite the fact that the number of particles A does not change when introducing particles B. From this point of view it would be interesting to recalculate the particle size distributions during boiling in order to eliminate the effect caused by hop compounds in order to analyze what happens to the medium and fine particles. As was mentioned for the heating phase the peak of the medium particles increased until start of boiling. Then, as boiling proceeded, in five out of seven boiling systems the peak level remained constant until end of boiling (exemplarily shown in Figure 9), while only in two cases the peak slightly increased toward the end of boiling (when coarse particle peak were eliminated by calculation). In conclusion, this means most of the hot break formation is completed already at the start of boiling and there is no considerable gain during the boiling phase itself. This finding might have far-reaching consequences for the boiling step. Since the precipitation of wort proteins is one of the aims of wort boiling as mentioned earlier, it is important to know when after the start of boiling this precipitation is completed. As the trials show, the precipitation mainly seems to be completed at the very beginning of boiling, that is, as soon as the whole batch of wort reaches boiling temperature. Therefore, according to these trials the boiling duration might be reduced to a minimum from



**Figure 7.** Sectional trends of particle size density distributions  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: most modern lauter tun (D); boiling system: thin film evaporator (D)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; turbidity during mash separation (broken line with points); temperature during wort collecting and boiling (broken line); highlighted area: heating/evaporation step prior to wort chilling. x axis: process step [left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: turbidity of lauter wort [EBC]/temperature during wort boiling [ $^{\circ}\text{C}$ ]; arrows indicate points of hop additions (hop 1 and 2: pellet hopping).

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the precipitation point of view. At least it may be concluded that protein precipitation is not the limiting step in terms of a process time reduction compared with the other technological goals of wort boiling.

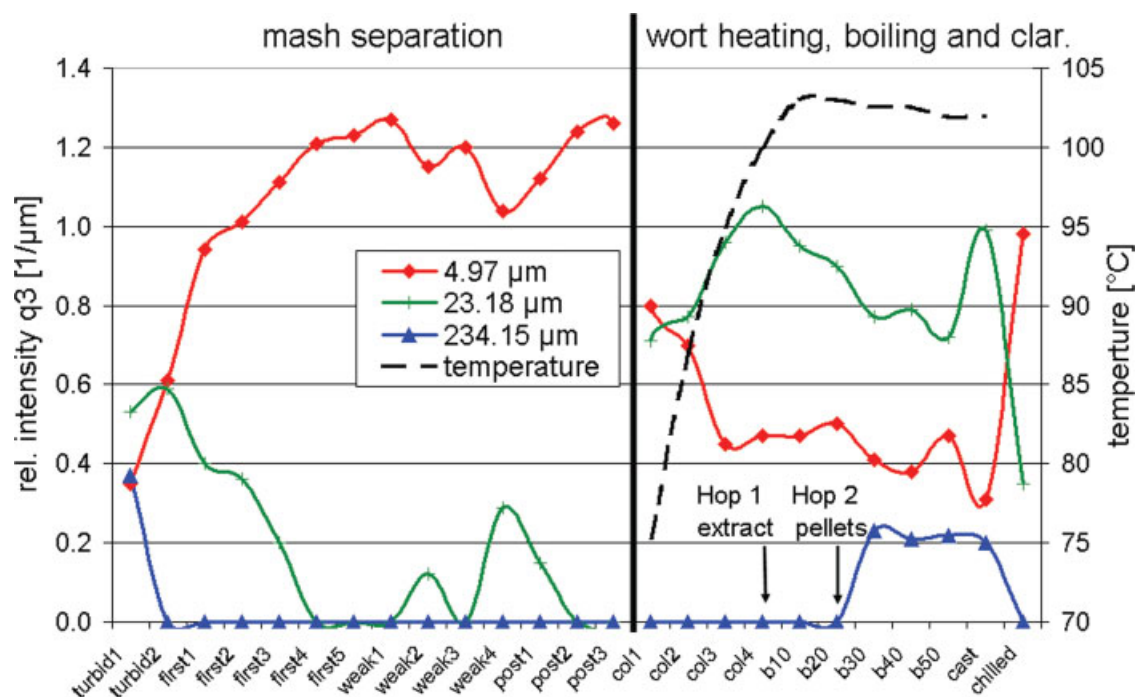
### Wort clarification

All investigated brewing systems used a whirlpool to separate the hot break according to the “tea cup effect,” which implies that the wort is tangentially introduced into the flat bottom vessel causing a rotation of the vessel content. This rotation causes the solid particles of higher density to concentrate in the bottom center of the vessel forming a cone of solids. The clarified wort is drawn-off from the outer diameter of the vessel. The aim is to provide a wort for the further brewing steps of fermentation and maturation, which is as clear and bright as possible for quality and technological reason. The sense of this intention is, however, currently under discussion.<sup>106,109,112</sup>

From the particle point of view the effect of the whirlpool step becomes apparent when comparing the wort composition at the end of boiling (cast wort) with that after whirlpooling and chilling (chilled wort). As can be seen from Figure 4 the coarse particles completely disappeared, the medium particles were slightly reduced, while the fine fraction definitely

increased in density distribution. This means, clarified wort mainly contained the fine fraction, which did not seem to be touched by whirlpooling. This is also valid for the medium fraction to a lower extent, while the coarse fraction is completely separated, if the whirlpool works properly. Thus, the remaining turbidity of post-whirlpool wort is mainly caused by the fine particles in the range of 5  $\mu\text{m}$  and only to a minor extent by medium particles of 20–25  $\mu\text{m}$ , but not by coarse particles of  $>200 \mu\text{m}$  in equivalent diameter. In terms of the particle size this result seems to be consequent since coarse particles are expected to sediment faster than medium and small particles, which could be confirmed here. Therefore, the measurement of particle size distributions allows to optimize the duration of the whirlpool stand since it may easily be adjusted to a preferred particle composition. Further, it seems to be possible to easily detect a lack or failure of the whirlpool function or any other wort clarification means by taking out particle size distribution measurements of cast wort and chilled wort. Correspondingly, when the formed hot break was analyzed the respective peaks at  $\sim 34$  and 234  $\mu\text{m}$  were observed.<sup>113</sup>

In the special case of the thin film evaporation boiling method, hot break is conventionally separated by whirlpooling but another heating and evaporation step, which is carried out



**Figure 8.** Sectional trends of particle size density distributions  $q_3$  of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (B); boiling system: external boiler (B)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; temperature during wort collecting and boiling (broken line); x axis: process step [left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; post = post runs (not collected); col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)]; y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: temperature during wort boiling [ $^{\circ}\text{C}$ ]; arrows indicate points of hop additions (hop 1: extract hopping; hop 2: pellet hopping).

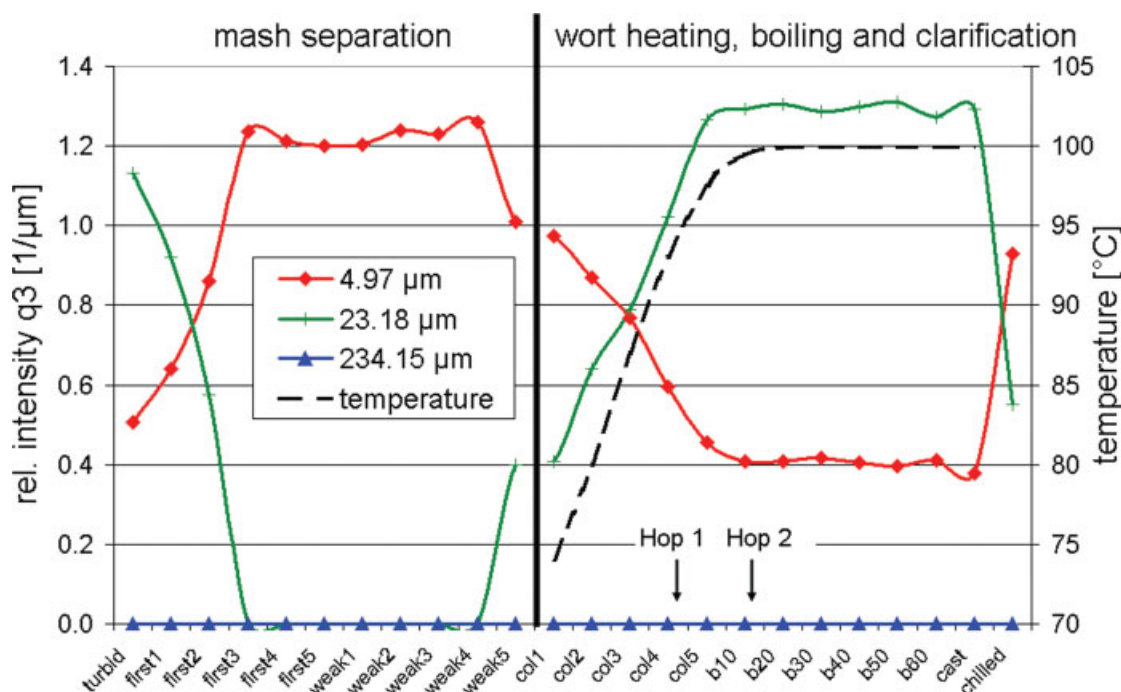
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at the end of the whirlpool procedure prior to wort cooling. This is done due to further remove the unwanted volatile aroma compound dimethyl sulfide, which is degraded from its precursor *S*-methyl methionine due to heat stress. As whirlpooling usually increases heat stress of the wort by keeping it at temperatures  $>90^{\circ}\text{C}$ , a significant amount of dimethyl sulfide is formed within this step and conventionally there is no way to reduce it thereafter. Here, the additional heating and evaporation step allows to reduce the dimethyl sulfide concentration directly prior to wort cooling with no further formation. Within this heating step it was observed that the fine particles rapidly decreased in favor of medium particles (Figure 7, marked box). Since lauter fractions and hop products can be excluded from influencing this step, this confirms the earlier hypotheses that heating leads to the formation of medium size particles.

Besides, as former and current investigations show the presence of particles of a certain size and composition seems to have a significant influence on the fermentation performance of the resulting wort. Therefore, the mash separation and particularly the boiling and clarification steps are important measures to influence size, number and composition of the formed and remaining particles present in wort as can be shown by applying particle size distributions and other methods.

### Technological parameters of wort boiling

The N coag. value represents the remaining coagulable high molecular nitrogen compounds in wort such as proteins and polypeptides. It is a key indicator for the non-biological stability (haze formation) as well as for the foam stability of the resulting beer and is therefore of crucial importance. Figure 10 shows the N coag. values in wort, wherein all values have been adjusted to an extract content of 12°P to eliminate dilution effects (e.g. which occur during wort collecting when weak wort is added to first wort). As can be seen from Figure 10, the N coag. values decreased with time no matter what boiling system was being used. More particularly, during wort collecting and heating, that is prior to start of boiling, a reduction was already observed, which continued during the boiling phase. This confirms the earlier assumption of hot break formation already taking place during heating up prior to start of boiling. In detail, the gentle boiling system (A in Table 3) and the thin-film evaporator (D) had high final values of  $>15$  mg/L, while the external boiler with post-stripping (C) and one conventional external boiler (G) had middle-range values of  $\sim 12$  mg/L. The internal boilers, either with forced circulation (F) or not (E), and the other external boiler (B) had low final values of 5–10 mg/L. This indicates that the conventional boiling techniques cause a strong reduction of N coag. while more modern



**Figure 9.** Normalized sectional trends of particle size density distributions of wort during mash separation, wort boiling, and wort clarification [mash separation system: lauter tun (F); boiling system: internal boiler with forced circulation flow (F)] representing selected particle size classes: 4.97, 23.18, and 234.15  $\mu\text{m}$  equivalent diameter; values normalized: particle signals of  $x_m > 50 \mu\text{m}$  have been eliminated by calculation (corresponding to non normalized diagram: Figure 4); x axis: process step (left: mash separation; right: boiling and wort clarification; turbid = turbid wort pumping; first = first wort; weak = weak wort; col = collecting/heating of wort; 1, 2, 3, etc. = sample no.; bn = boiling, sampling  $n$  minutes after start of boiling; cast = cast wort (prior to whirlpool); chilled = chilled wort (after whirlpool)); y1 axis: relative intensity  $q_3$  [ $1/\mu\text{m}$ ]; y2 axis: temperature during wort boiling [ $^{\circ}\text{C}$ ]; arrows indicate points of hop additions (hop 1 and 2: pellet hopping).

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systems seem to be gentler in effecting in protein coagulation. Since the particle size distribution measurement does not allow to quantify the number of particles of each class, it seems to be difficult to correlate the N coag. reduction and the particle sizing in terms of quantification, however, both methods indicate that coagulation reactions take place already prior to boiling.

While the TBAN depends on the quality of the raw materials used for brewing, its increase from the start of boiling (dTBAN) is independent from raw material effects and therefore a well-known heat load indicator of wort. As can be seen from Figure 11, the TBAN increased for all systems during boiling and further during wort clarification due to the whirlpool stand at temperatures of around  $95^{\circ}\text{C}$  or above, where the formation of TBAN still takes place. When comparing the dTBAN at the end of the particular boiling procedure (see Table 3) three groups may be identified: dTBAN is very low and in the range of 5–10 for the external boiler with post-stripping (C in Table 3) (due to pre-cooling of wort<sup>97</sup>), the gentle boiling system (A) and the thin-film evaporator system (D). The external boiler (G) was in the range of around 11, while the other external boiler (B) as well as the internal boilers (F, E) had values of 17–19. This correlates well with the findings for N coag.: the rougher the boiling the higher the TBAN increase and the greater the N coag. decrease. An increase in TBAN

was already observed during heating of worts before boiling temperature was reached.

## Conclusions

From the results of the large and pilot scale trials undertaken in this investigation it may be concluded that three basic different particle sizes occur in wort in the steps of mash separation, wort boiling and wort clarification: fine (ca.  $5 \mu\text{m}$ ), medium ( $20\text{--}25 \mu\text{m}$ ) and coarse particles ( $>200 \mu\text{m}$ ). During mash separation a high wort turbidity is characterized by appearance of medium sized particles, while in clear wort fine particles are dominant and medium and coarse particles are absent. Toward the end of mash separation, when EBC turbidity increases again due to raking, medium and even coarse particles may be present in wort, depending on design and age of lauter equipment as well as process control characteristics. This means different kinds of particles seem to be responsible for different levels of wort turbidity. Thus, the design and process control of the lauter system have an influence on the composition of the resulting wort, not only in terms of turbidity but also in terms of particle composition. It may be concluded that the particle size distribution correlates to the EBC turbidity to a certain extend. It is the disappearance of medium and coarse particles

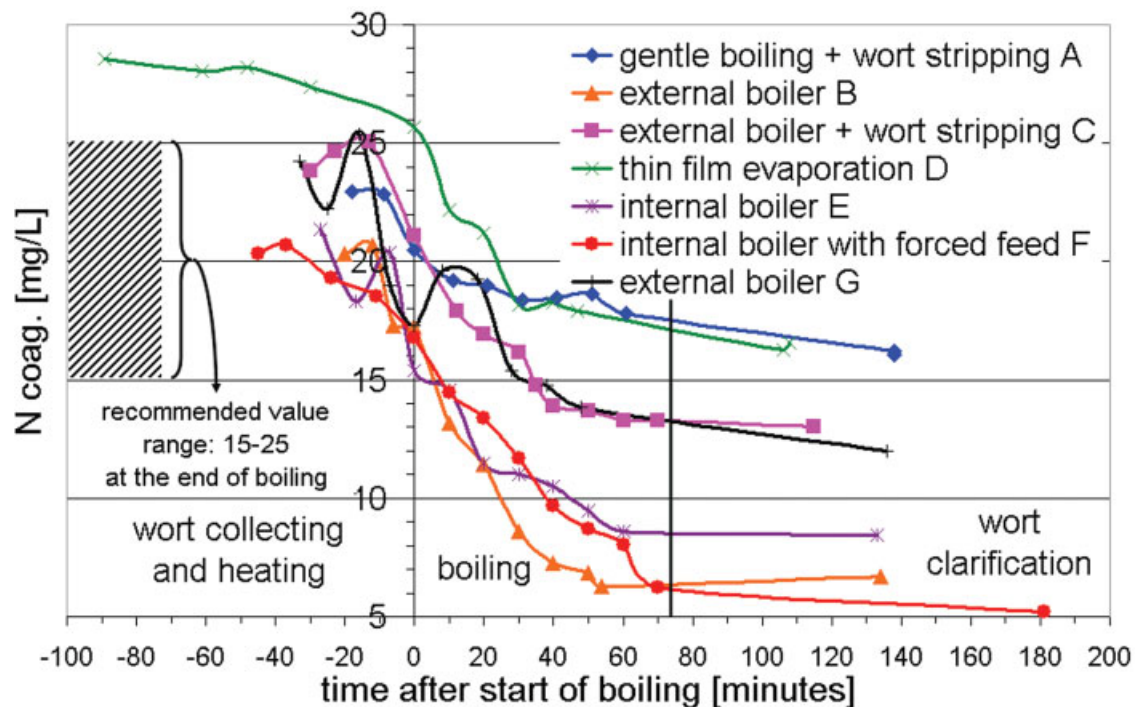


Figure 10. N coag. values in wort during collecting, heating, boiling, and clarification of wort; variation of boiling systems according to Table 3; values normalized to an extract value of 12°P to eliminate dilution effects; hatched area: recommended content in cast wort according to Table 1.

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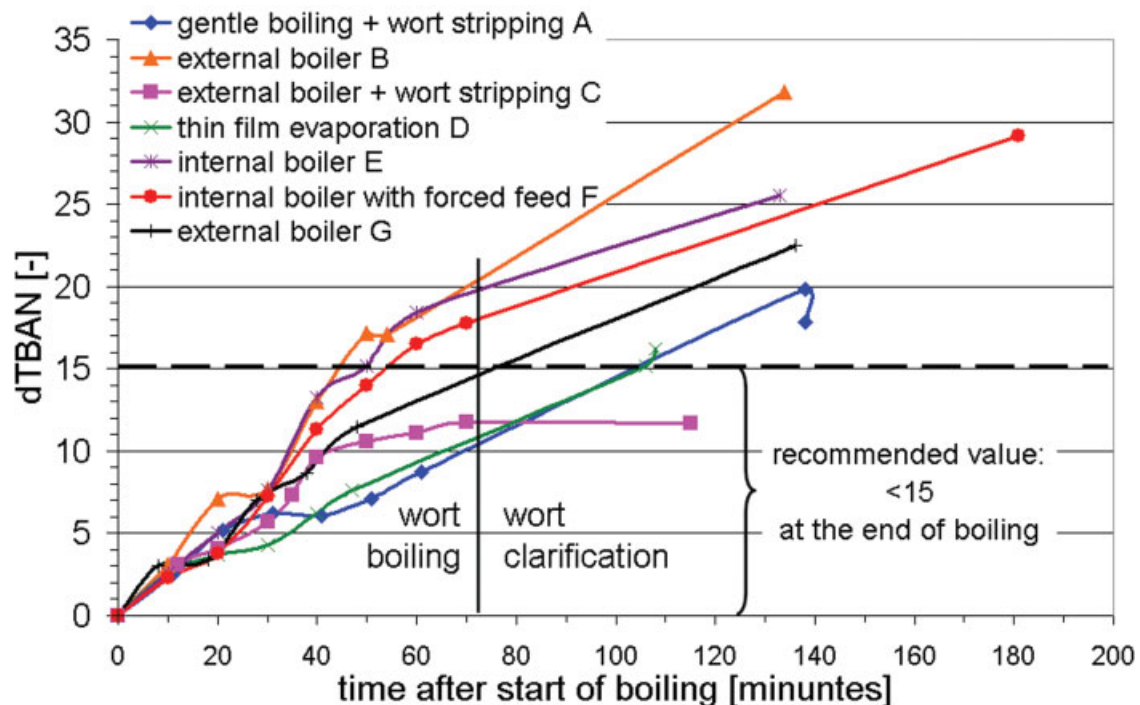


Figure 11. Increase of TBAN values (dTBAN) in wort during boiling and clarification; variation of boiling systems according to Table 3; broken line: maximum recommended increase in cast wort according to Table 1.

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that cause a decrease in EBC turbidity. While lauter tuns usually eliminate medium and coarse particles when running clear wort, the mash filter investigated here removed coarse particles completely, but medium particles only partly.

The process of wort boiling may be divided into wort collecting/heating phase and the boiling phase itself. During heating of wort to boiling temperature a coarsening of the particles in favor of the medium particles occurs. This is due to the formation of hot break already during heating up of wort, which is indicated by a reduction of N coag. levels and an increase of TBAN already prior to start of boiling. It is also shown that the way of hopping (pellets or extract) plays an important role in terms of the particle composition of the resulting wort. The addition of hop pellets causes signals of coarse particles (>200  $\mu\text{m}$ ), which can easily be distinguished from hot trub particles. In contrast, if soluble hop extracts are used, these coarse particle peaks do not occur. If the coarse signals are eliminated by calculation it is observed that the ratio of medium to fine particles is constant throughout the boiling phase. This means most of the hot break formation takes place at the begin or even prior to boiling, while toward the end of boiling not much change is observed in terms of particle size distribution. Further, at wort clarification using a whirlpool the coarse particles are completely removed from wort, while medium particles are reduced, both due to hot break separation. Thus, applying particle size measurement allows to evaluate a proper whirlpool function. If this solid separation step is working properly, the clarified wort mainly contains fine particles similarly to a clear lauter wort.

These investigations show that the process steps of mash separation, wort boiling, and wort clarification can be characterized by particle size distribution measurements, which allow to gain further technological information in addition to the conventional turbidity measurement. Changes in chemical analytes such as N coag. and TBAN partially correlate with changes of single particle fractions. Furthermore, in practical brewing operations particle size distribution measurements may be used to monitor if single process steps such as mash separation or wort clarification work properly.

Since this investigation gives an extensive overview on the influence of different technologies applied in the brewing process on the particle size distribution of wort throughout the brewhouse procedure, it seems to be promising to undertake multiple trials for each of the investigated system in order to statistically confirm the observations made within this investigation. Further, it seems to be interesting to isolate the particles that cause the respective peaks in the size distribution and to identify their composition in future trials in order to learn more about the brewhouse process steps and their effect on the fermentation process and the resulting beer quality. In the near future, the measurement of particle size distributions of wort might be used to optimize the investigated process steps by tailoring the most effective quality and quantity of particles for an optimum fermentation performance and beer quality.

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