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Measuring Particle Size Distribution of Mash with Laser Diffraction to Evaluate the Process Success

The coherence of particle size distribution and reaction behaviour is well-known in process engineering as well as in the brewing industry [15, 17, 21]. Additionally, the particle size distribution can give precious information about the status quo of several processes. Usually, in the brewhouse, only the particle size distribution of the malt grist is measured by sieving [4]. But, the particle size distribution changes during the mashing process. To measure these changes, wet sieving methods are possible but too complex to get fast and authentic results. Thus a fast and reproducible method is missing for mash. Therefore, an analysis method with laser diffraction was developed, which give brewers now the possibility to analyse the status of the mashing process. Against the background, this analysis is not applicable in every brewhouse, a method was investigated to freeze the samples and analyse them in the laboratory. With this new knowledge, now it possible to find out correlations between the particle size distributions, mash conversion, viscosity and filtration performance.

Descriptors: particle measurement, milling, mashing, lautering, brewhouse

1 Introduction

Increasing costs for energy, small capacities in the brewhouse and variable qualities of the raw materials – these and other arguments are factors which ask for solutions to prevent losses and problems during beer production. While most of the technological impacts are well investigated, many procedural impacts on the beer production process are still unexplored. An increasing number of developments of machines, for the mashing process as well as for the lautering process, show the big interest of new ways to find out the best possibility to produce beer, especially in mashing [2, 5–7, 12, 13, 19]. As Stieß mentions, it is only possible to optimize processes if the disperse status and behaviour are able to be described [18]. This motivation is an additional aspect to learn more about the procedural characterisation of the suspension mash.

The influence of the particle sizes distribution of the malt grist is a well-known and very important factor for the successful process of mashing and lautering: finer grists are followed by higher yields, but can also lead to serious problems during the lautering process in the classical lauter tun [15, 17, 21]. To find out new ways and solutions, a couple of investigations on the particle technology in the brewhouse were already done in combination with further analysis [1, 8, 24].

To find out the best grist composition, the molinology usually uses a plan sifter (Pfungstätter Plansichter) to analyse the ground stock [4]. But this is primarily possible for dry milling processes. For the control of the particles in wet mills, methods of wet sieving are typically used. An exact rating of the particles and the classification of a specific particle size range is very complex [3]. Especially influences like ultrasonic treatment or shaking can lead to false results because of effects of agglomeration or desagglomeration. A fast and reproducible measuring system is not established up to now.

The ranges of particles, which can be measured, depends on the range of the distribution in the disperse system and the measurement system [11, 16, 18]. Therefore, before the decision on a certain measurement system is done, the range of interest of particle sizes has to be defined. Mash is a very complex suspension with particle varieties from nano to millimetre particles, whilst the most interesting fraction in the mashing process is settled between 5 and 500 µm because of the size of the starch particles and the agglomerates [15, 16].

The first measurements in this work were done with a standard analysis method, given by Sympatec and often used for disperse suspensions. But in contrast to mining or chemical products (e.g. insolubility of sand, stones, minerals or plastic particles), mash is a biochemical suspension. This means, that a very careful and fast analysis of the samples is essential.

First analyses, done with the standard analysis method, showed non-satisfying results with scientifically non-discussable results (comp. Fig. 1).

These results were the activator to find out the best analysis method for analysing the particle size distribution in mash.

2 Materials and Methods

2.1 Mashing

For the production of the experimental mash, a laboratory mash tun (Laboratory Reactor, IKA, Staufen) with a filling volume of

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Figures see Appendix

2.2 l was used. Standard malt (IREKS Pilsener style) was mashed with a standard mashing regime (comp. Fig. 2). The mash was produced with 500 g of ground malt and 1800 ml of water.

2.2 Particle Measurement Device

The particle analysis was done with a HELOS Laser Diffraction spectroscope, Sympatec, Clausthal-Zellerfeld (Fig. 3).

The spectroscope can be used for dry and liquid disperse systems. The wet samples were analysed in a cuvette. The lense used for the analysis had a measurement range from 4,5–1750 µm.

The measuring principle is shown in Figure 4.

2.3 Standard measuring specification for a liquid HELOS analysis (not applicable for mash)

The preparation of the samples for the analysis in the laser diffractor is essential. The standard measuring specification for liquid disperse systems was given by Sympatec. After degassing the analysis cuvette by pumping, the sample is given in to the cuvette up to an optical concentration between 5 and 15 %. Before the measurement, the sample is degassed again with an ultrasonic device in the cuvette do expel the last remaining gas bubbles which could influence the result. After that, the measurement can be executed. The results are transferred in to the analysis software.

The HELOS used for the trials is a standard measurement equipment of the institute which is audited in regular intervals with Sympatec SiC reference material particles.

2.4 Sampling for instant analysis

Samples of the mash were taken with a specifically constructed sampling device, a piston with a 7 mm pipe. The volume of the samples was 10 ml. After taking out the mash, a sieve cut with a mesh size of 1000 µm was done immediately whilst the filtrate was given in to cold water.

2.5 Sampling for analysis after a frozen storage

10 ml samples were taken out and given in to a small plastic vessel. This was shock-frozen with liquid nitrogen and stored at –22 °C for later analysis.

2.6 Microscope Analysis

The microscope mashing and picture analysis was done in a 15 ml microscope mash tun (comp. Fig. 5).

The mash was heated with the same regime as in the 2.2 l kettle. The mash was diluted down to the best optical concentration for a microscope analysis.

With a digital camera and the software a4i-Docu 5.0, Aquinto AG, pictures were taken at the same time when the 2.2 l-mash was sampled. The picture analysis for the starch particle sizes was done with the software Optimas 6.5 1, Weiss Imaging and Solutions GmbH. More than 100 starch particles were observed and analysed to gain a sufficient number of data. The analysis software was calibrated with standardized glass particles and a calibrated glass slide with calibration scale of 100 µm (comp. Fig. 6).

2.7 Statistical evaluation of the results

For the correct discussion of the results, a statistical analysis method had to be used. Considering the shape factor of the particles, a number of 5 measurements was defined. The calculation of the confidential intervals was done with the Student's t-distribution with a level of significance of 95%.

2.8 Defining the measurement specification for mash analysis

The experimental series were done in dependence of various test and analysis parameters. As important influencing factors, the following points were investigated:

- influence of sample stocking and storage temperature
- influence of analysis temperature
- influence of ultrasonic
- influence of analysis time

3 Results and Discussion

The measurement with the recommended measuring specifications (e.g. use of ultrasonic for degassing) generated unfeasible results (comp. Fig. 7). The enormous confidential intervals make the results senseless and scientifically not meaningful.

A long storage time of the samples at the wrong temperature also affects the results adversely.

Considering this fact it would be the best, if the probe is measured immediately in the cold state. But this is not possible because of the cuvette getting fogged up because of the temperature difference. It was found, that a dilution to the best optical concentration between 5 and 15 % should be done shortly before measurement at a temperature of max. 15 °C.

Satisfying results with reliable confidential intervals were gained after the development of the new analysis scheme.

Several cross-checks lead to a measuring scheme like shown in figure 11. It was both checked, for fresh samples as well as for shock-frosted samples. One result of the measuring of the same samples, once fresh, the other frozen, is shown in figure 10.

The influencing parameters, storage time at warm temperatures and ultrasonic degassing shortly before the measurement, showed big effects on the meaningfulness of the results and, as it was shown, delivers useful results. Figure 9 gives an example of one of the results, achieved with the new developed analysis specification.

As clearly visible, the confidential intervals are rather small, which documents the repeatability and significance of the results. In the area of the smaller and bigger particles, the confidential intervals are slightly bigger than in the middle size area. Reasons for that are agglomerates of starch particles and other particles. These agglomerates do not have the ideal spherical structure thus variations are measured in dependence of the direction of the particle passing the laser light in the cuvette.

There are two main reasons why the measuring scheme of the supplier did not work with mash from the brewhouse. The ultrasonic wave change the structure of the agglomerates in the mash thus a secure measurement is not possible after this treatment. Another point, why the suggested measuring system did not work is the fact, that mash is a biochemical suspension. As commonly known, biochemical reactions depend on temperature and increase their activity with increasing temperatures [9]. Thus, the use of 0 °C cold water is the best for storage of samples until analysis. To avoid fogging of the cuvette as well as unwanted changes in the solution, the dilution of the probe to the best optical concentration should be done shortly before the analysis.

In most of the cases, a particle analysis with HELOS laser diffraction is not possible on-site. The samples for analysis have to be stored as mentioned above in the freezer. It is known that freezing leads to unwanted hazards [10], but it was found out that the influence was negligible in comparison to a warm storage (> 20 °C and > 30 minutes till analysis).

There is a wide range of measuring systems to analyse the particle size distribution of suspensions, but not every system can fulfill the demands of mash. Next to time for analysis, the particle range which should be analysed was the main criteria for the used system.

The system HELOS is, as previously mentioned, the standard measuring device for the work of the chair of process engineering. In the industry, there are several further methods. It is the nature of the science of particle size measurement that comparisons have to be done very conscientious to make no misinterpretations on absolute values.

The information which can be gathered with the particle size analysis of mash is the status of the mash in dependence on the particles situation.

As it is well known the starch granules are growing during the mashing process, this might be the cause of the changes of the particle size distributions during the mashing process. To support this theory, the starch particles were analysed under a microscope.

The comparison of figure 12 and figure 13 shows a difference because the measuring range of the two systems was different. But the non-absolute values show the same results. The particles are growing with the same behaviour during the mashing time. For the main questions, whether the HELOS analysis system is feasible for a judgement and characterisation of mash, these gained results underline that.

4 Conclusion

The main scope of the investigation was to develop a measurement system which allows suppliers and brewers to judge the mash in correlation to their particle size distribution. The development was started when mash samples were taken, frozen and sent to a laboratory which measured the probes according their standards.

A closer look showed that the biochemical degradation of the probes has to be stopped and the sensitive suspension has to be analysed very carefully.

The new analysis method was applied for the same trials as in the beginning, when the results were useless. The results had a good confidence and showed interesting correlations. The first trials were in coherence to the filterability.

Since the development, the measuring system was used for numbers of measurements at the institute. In fact, it is an important tool now and was used for a couple of investigations. It helps to understand the desagglomeration process of the ground malt much better, it gives the possibility to follow the gelatinization of the starch in a new way and, as a big chance for the future, it delivers basic knowledge for the development of an optical process control system as a new way of mash control [20, 23, 22]. The use in the case of milling, filtration and quality of malt is investigated at the moment.

5 Literature

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Appendix

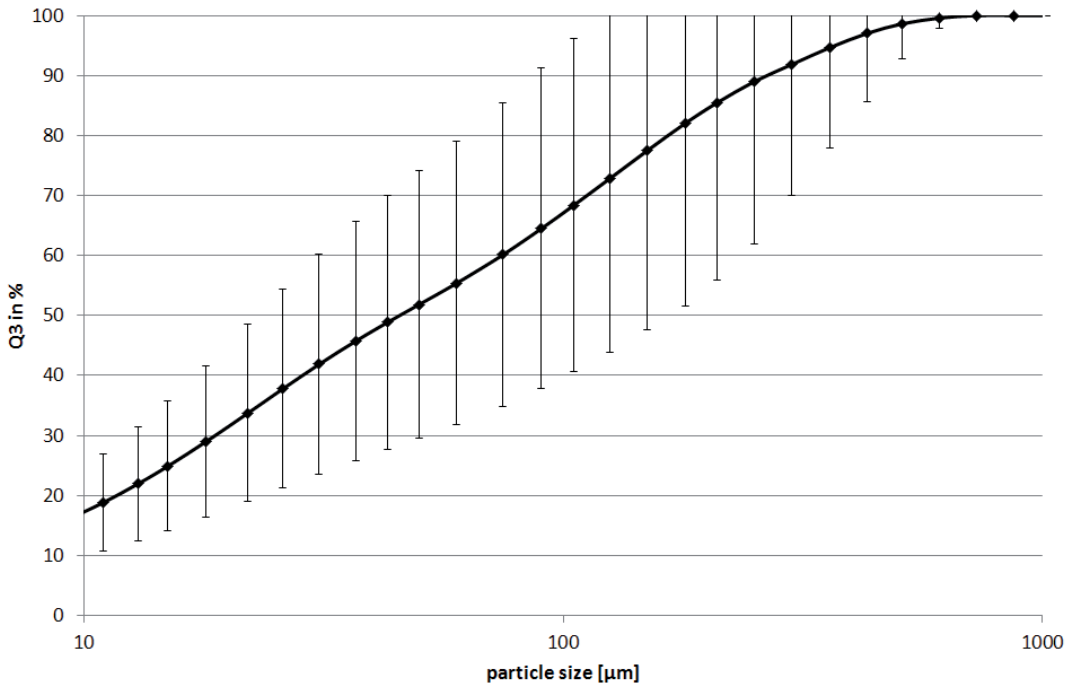

Fig. 1 Example for the result of a particle size measurement (triple determination), done with a standard analysis method which was not applicable for mash

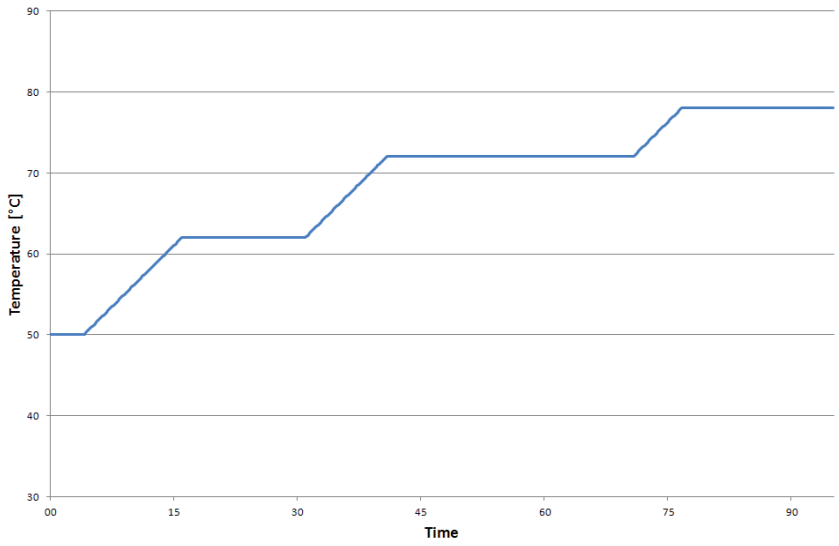

Fig. 2 Standard mashing regime, used for the trials in the 2.2l-reactor and the microscope reactor

Fig. 3 The HELOS laser diffractions spectroscope, used for the development of the analysis method

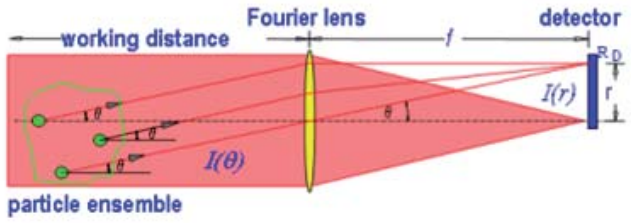

Fig. 4 Working principle of a laser diffraction spectroscope [14]

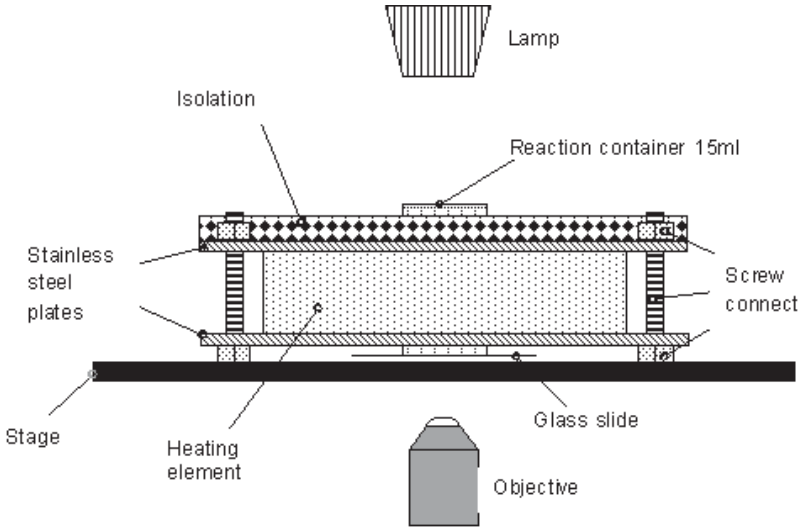

Fig. 5 **Microscope mash tun, integrated in a microscope**

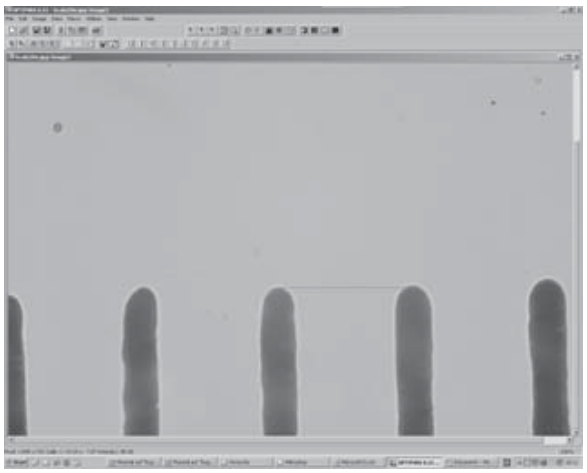

Fig. 6 **Calibration scale on the glass sheet, calibration distance 100 µm**

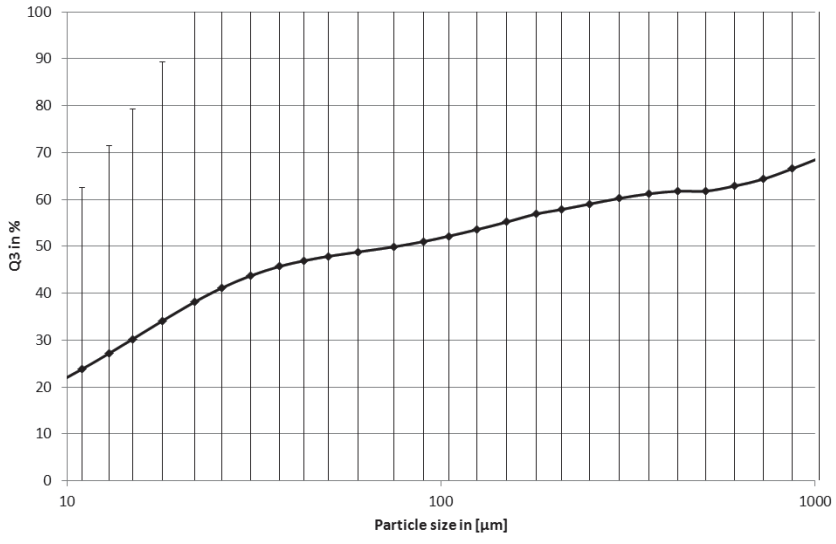

Fig. 7 **Q3-distribution after treatment of the sample with ultrasonic like in the standard measuring specification of the supplier**

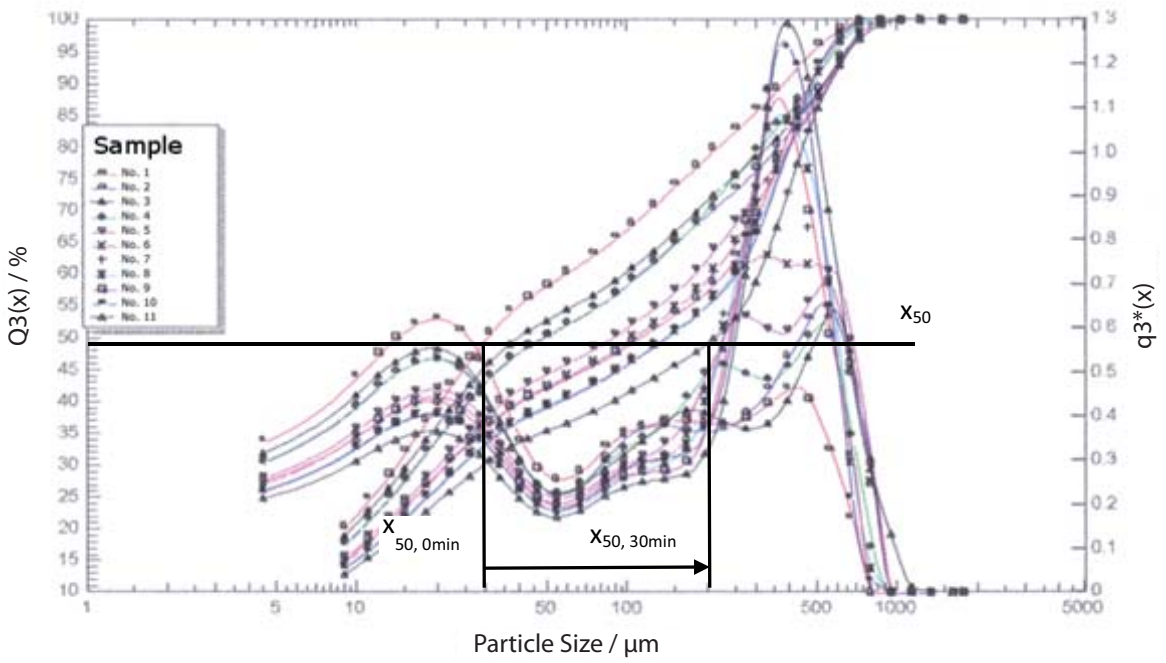

Fig. 8 **Screenshot of the measurement series 30 min at a constant temperature of > 20 °C, measuring every 3 min, with a significant change of the x_{50}, from 30 µm to 200 µm**

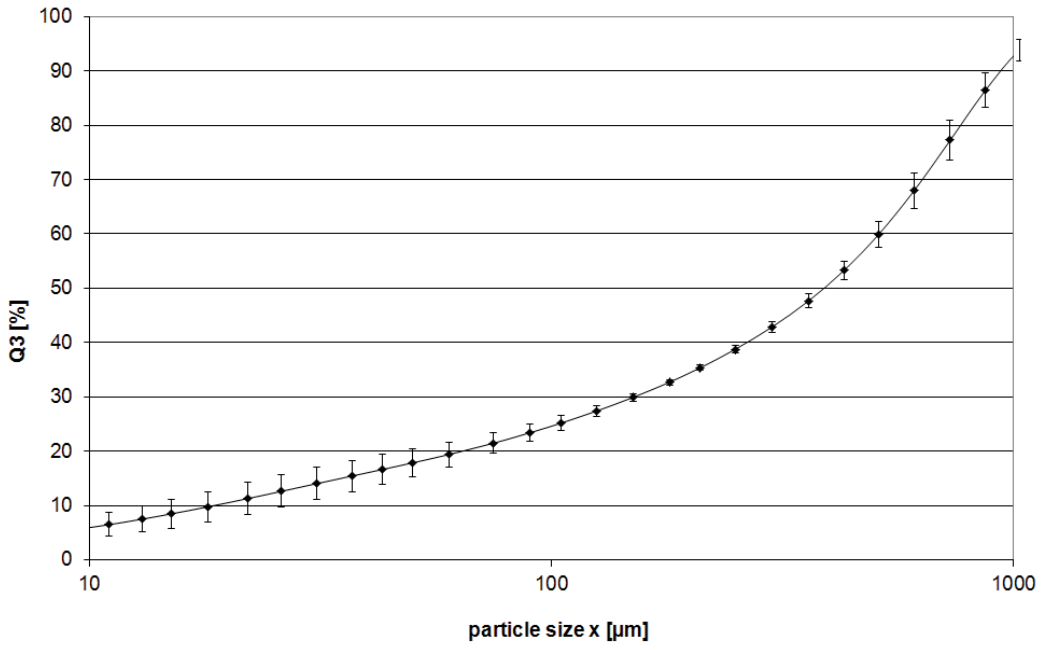

Fig. 9 **Q3-distribution of a mash sample, which was analysed with the new analysis scheme**

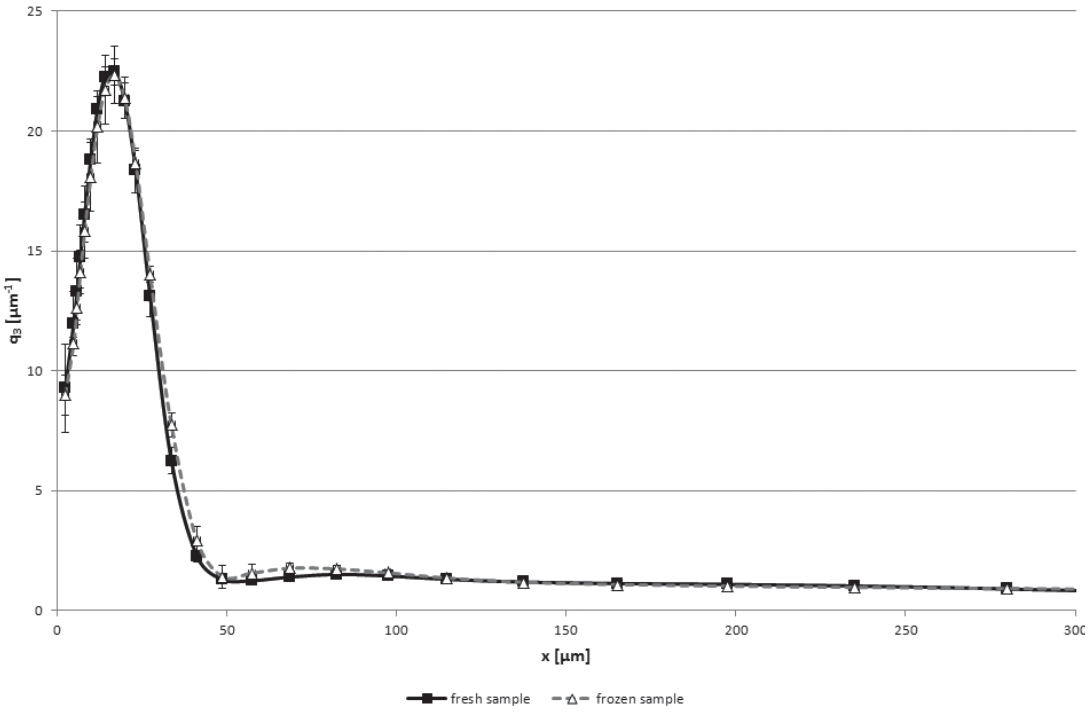

Fig. 10 q3-distributions of samples from the same mash, before and after freezing

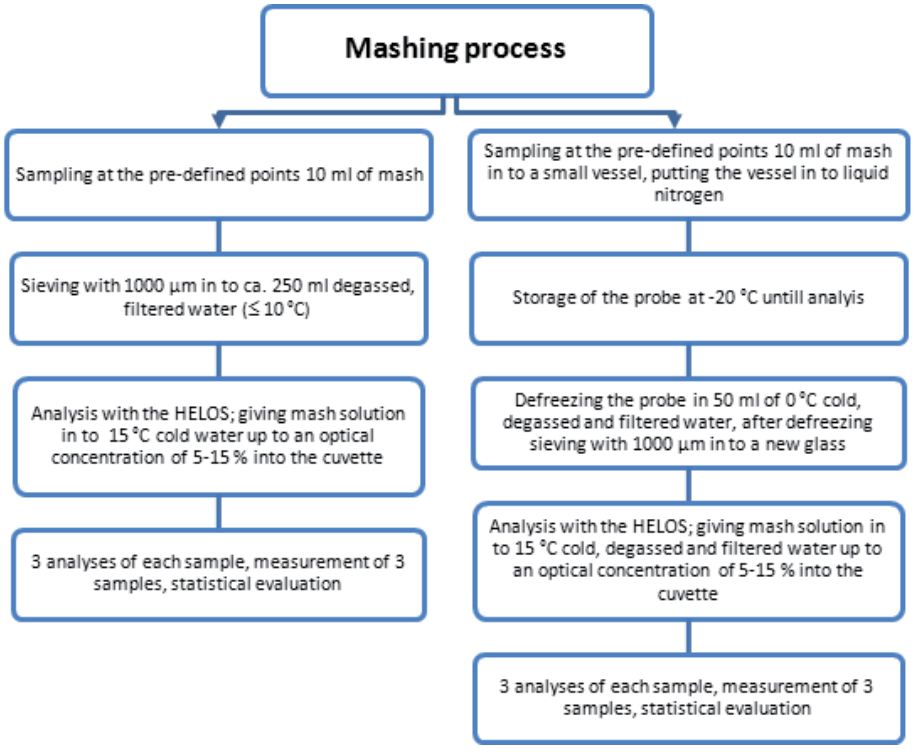

Fig. 11 Flow chart of the analysis of particle size distribution of mash

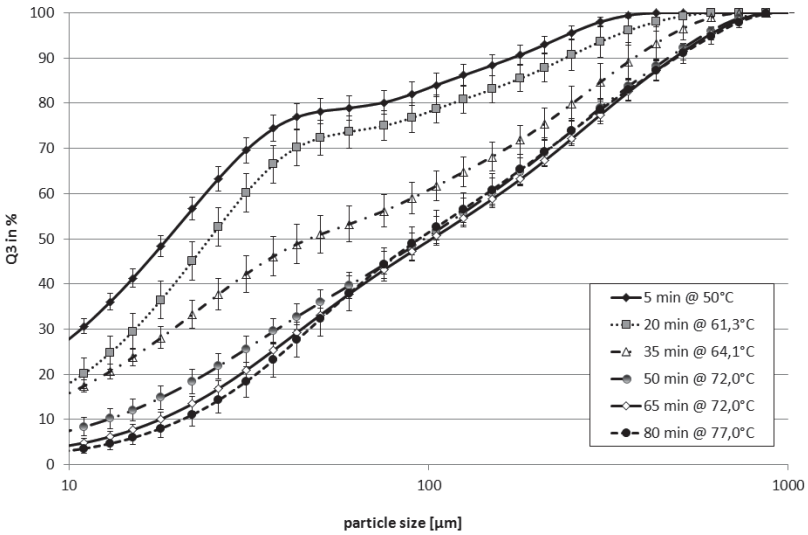

Fig. 12 **Q3-distributions at different sampling points, measured with the HELOS system and a significant change of the x_{50} during the mashing time**

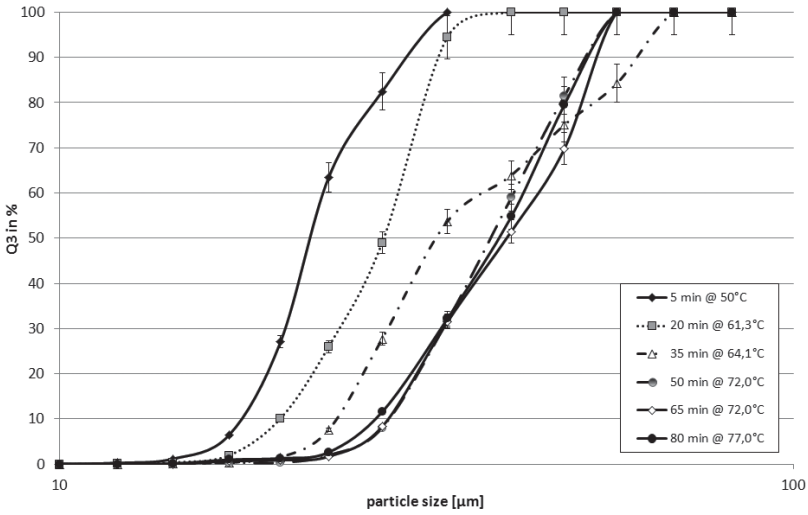

Fig. 13 **Q3-distributions at different sampling points, measured with the microscope and a significant change of the x_{50} during the mashing time**