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ORIGINAL PAPER

An accurate approach to assessing the hop particle size and its filtration behavior during dry hopping

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Abstract
The hop pelletizing is not only influenced by the hop variety and the associated α-acid contents, but also by many annual and regional variations. In this context, the particle size distribution of the hop products had a subordinate role due to its main application in the brewhouse with its principle focus on α-acid content. But due to a high use of hops for dry hopping and associated large beer losses along with the separation of the hop residues, hop particle size is becoming more important. Thus, the aim of this study was the development of a method determining particle size distribution of hop products (powder and pellets) to assess its impact on filtration performance. Using citric acid phosphate buffer (pH 4.5) with 5 vol% ethanol as a solvent resulted in a similar particle size distribution as in beer. In addition a swelling and disintegration time of 60 min was determined for all hop products. The particle size measurement had an average standard deviation of 25.9 µm ($Dx(50)$, $n=180$) with a confidence interval of 3.78%. The comparison of average particle size ($Dx(50)$) and filtrate flux resulted in a significant correlation ($r=0.846$, $P<0.001$, 58 investigated hop products), whereas hop products with a higher average particle size had a higher flux. Therefore, the method allows the investigation of hop products for the use in dry hopping to achieve optimal filtration performance and low beer losses. Furthermore, the pelletizing process can be examined to produce separate products for application in brewhouse or fermenting and storage cellar.

Keywords Beer · Brewing · Dry hopping · Hop pellets

Abbreviations

HEB	Hersbrucker Spät
HMG	Hallertauer Magnum
HTR	Hallertauer Tradition
HKS	Herkules
E	Elbe-Saale (growing region)
T	Tettnang (growing region)
PLA	Polaris
PER	Perle
HAL	Hallertauer Mittelfrüher
MBA	Mandarina Bavaria
H	Hallertau (growing region)

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Introduction

Hops are also known as the "soul" of beer, even if they are traditionally used only in low concentrations. Nevertheless, they do not only impact flavor and bitterness, but also increase the microbiological stability of the beverage [1, 2]. However, due to problems occurring in a technical environment when using whole hop cones, today mainly hop pellets are processed in breweries. This has not only the advantage of better packaging and storage (no susceptibility to oxidation), but also a simplified dosage in beer production is possible. They also cause less wort and beer losses due to a lower spent hops volume [3–6]. Furthermore, a better homogeneity of the hop products itself can be ensured over a longer period of time [5]. In addition, a facilitated mass transfer of hop constituents into wort and beer due to a higher surface by ruptured lupulin glands can be observed. Nevertheless, because of an enrichment of the constituent components in hops during pelletizing, it may also lead to the reduction of undesirable substances such as nitrates or pesticides (only in type 45 pellets) which are associated with the leaf material [1, 7].

The pelletizing process is complex and depending on the desired pellet and composition, different processing parameters are used over the world. The main pellet types are the conventional pellet type 90 and the lupulin-enriched type 45 [1, 8]. Depending on the desired pellet types, processing procedures vary with drying, crushing (milling types), sieving, processing temperatures as well as pressing of pellets. Due to the low purification, the conventional type 90 pellet differs only slightly from dried whole hop cones. A reduction in water content to 8–10% at temperatures below 60 °C results in about 95 kg of conventional pellets from 100 kg of hop cones. After grinding by means of a mill and homogenization in a mixer, the powder is pressed through dies with a defined cylindrical channel geometry. The applied pressure and shear forces in the dies result in a rupturing of the lupulin glands. Since the ingredients of lupulin are very susceptible to oxidation, the pellets must be cooled quickly and packed under inert gas [1, 5, 9–13].

In comparison, for the production of lupulin-enriched type 45 pellets, the whole hops are initially cooled to about − 35 °C and crushed in a cutting mill. Freezing of the cones solidifies the sticky hop resin fraction and oils inside the lupulin glands, which enables sieving and homogenization of the fine materials. The coarse leaf materials were sieved several times to increase the lupulin yield. The highest possible proportion of lupulin is the process objective of this pellet production [1, 11].

Since most knowledge on the pelletizing process is empirical and influenced by differences in the varieties (mainly by the α-acid and oil content, respectively) as well as many annual related variations, only little information is available on how the pelleting process affects hop particle size. Particle size of the hop products was so far of minor interest, caused by the intended use of hop pellets primarily in the brewhouse with a main objective of high α-acid yield and flavoring substances when used in the whirlpool [14–16]. But due to the use of high amounts of hops by craft brewers, new fields of application have been expanded with new requirements on hop products [17]. This so-called dry hopping can be performed with different process technologies like dosage in a fermenting or storage tank or filtration with special equipment [18]. Nevertheless, hop leafs have to be separated from the beer at the end of reaction time which is a key problem of this brewing methodology [18–21]. Problematic factors are large beer losses due to a swelling of the hop material and a poor separation of the solid hops ingredients as well as an insufficient hop aroma yield [18]. This can be controlled by the dry-hopping technique applied, the application period and the particle characteristics of the hop products. For this purpose, both filtration and sedimentation processes can be used. Both are dependent on the particle size of the hop constituents.

Due to a lack of analytical methods, the impact of hop particle size distribution as well as the disintegration and swelling of the hop pellets and powders on filtration behavior during dry hopping has barely been studied. However, a possibility of prediction would offer opportunities to optimize the use of hop products in the cold area of the brewery. For this reason, the aim of this study was the development of an accurate method for the investigation of hop products with respect to particle size and filtration performance to select hop products with a high filter performance. The established method will be applied to different hop samples to investigate correlations between the particle size and the filter performance.

Materials and methods

Materials

Hop products were purchased from different manufacturers all over the world. In total, 58 hop samples (10 hop powders, 34 type 90 and 14 type 45 pellets) were analyzed. Hop samples were stored at 0 °C after opening until all analysis was done to slow down oxidation processes.

Determination of particle size

Before the particle size distribution of the hop products could be analyzed, various influencing factors such as the optimal solvent, the swelling and disintegration time and sample preparation had to be investigated.

Selection of a suitable solvent

To determine the hop particle size, hop products must be dissolved in a suitable solvent, which is necessary to extract and disintegrate the particles from the hop pellets without damage. The solvent used should be comparable to beer. However, filtered or unfiltered beer was not suitable as a test medium because of its impact on the particle size measurement due to its varying composition (e.g., haze particles) and the contained carbon dioxide. Thus, the impacts on particle size distribution and filtration were tested with different solvents and compared to degassed filtered lager beer (decarbonized via vacuum pump). Besides distilled water, citric acid phosphate buffer (pH 4.5—McIlvaine buffer) was tested. In addition, the influence of ethanol on the measurement should be determined, which is why 5 or 10 vol% ethanol was added to the buffer.

Citric acid phosphate buffer (pH 4.5) was produced from a solution A, containing 21.01 g citric acid monohydrate in 1 L distilled water and a solution B containing 71.62 g disodium hydrogen phosphate dodecahydrate (Sigma Aldrich) in

1 L distilled water. For 1 L buffer solution with a pH of 4.5, 454 mL of solution B was filled up to 1 L with solution A. Subsequently, the pH of the buffer was checked.

Sample preparation

For the particle size measurement 5 g/L hop product was weighed into bottles and filled with 200 mL solvent.

Swelling of hops

Since one particle measurement needed up to 5 min, a swelling of the hop products and their impact on the particle size distribution must be investigated. Furthermore, particles must be disintegrated from pellets without damage before a measurement is possible. For this reason, the particle size of various hop pellets was first determined within 1 h in a 10-min cycle and then within 3 h in a 30-min cycle. To show significant differences of the swelling time on hop particle size, the mean particle sizes *Dx*(50) were examined by one factorial ANOVA (Tukey test). The influence of swelling is shown in Fig. 2. An optimal swelling and disintegration time of 60 min at room temperature could be determined for all hop products, which was used for all further measurements.

Measurement of particle size distribution

Particle size distribution was analyzed using Mastersizer 3000 (Malvern Panalytical GmbH, Kassel, Germany). This laser diffraction particle size measuring device determines the scattered light intensity of a laser beam as it passes through a particle sample. Small particles lead to large scattering angles, while large particles scatter light at small angles. According to the manufacturer, particles with a size of 0.01–3500 µm could be determined [22]. The results are given as volume fractions with respect to the particle size. This evaluation gave the best results for the filtration compared to the alternative number and surface distribution.

The hop pellets were solved in McIlvaine (pH 4.5, 5 vol% ethanol) 60 min before their particle size distribution was measured via Hydro EV wet dispersing unit. For each sample, four series of measurements with five repetitions were performed ($n = 20$). For each series of measurements, 25 mL of the sample was added to the dispersing unit. All samples were homogenized by shaking before measurement.

The following settings were used for the measurement: blue light measurement duration, background measurement duration and sample measurement time were 10 s. The shading limit was set between 0.1 and 20%. The sample dispersion was ensured by means of a stirrer (2300 rpm). Data analysis was performed with a universal model using a particle type of optically opaque particles (Fraunhofer approach) in the dispersing medium water (refractive index: 1.33).

Microscopic investigation of particle size

Because Malvern Mastersizer allowed only a particle size measurement up to 3500 µm, a microscopic method to determine bigger hop particles was used. Particles were photographed with a VHX digital microscope (KEYENCE DEUTSCHLAND GmbH, Neu-Isenburg, Germany) at 150 × magnification and then investigated by image analysis software. 300 µL sample was transferred on a microscopic slide and five images were taken with a resolution of 8192 × 6595 pixels. Image analysis was performed with Image J (developed at the National Institutes of Health by an employee of the Federal US Government), a Java-based image processing and analysis tool. The image was converted into an 8-bit type and scale was set at 0.694 pixels/µm. Dark outliers with a radius smaller than 5 pixels and a threshold smaller than 50 pixels were removed.

However, particle analysis with this microscopic method has various influences such as the overlay of hop particles and the non-homogeneous distribution of the sample. In addition, particle size can only be calculated using the diameter of an assumed spherical shape of the hop particles, which results in further deviation. For these reasons, microscopic analysis of hop particles was only used to study outliers and very coarse hop samples.

Filtration trials

To investigate the filter performance of hop pellets during dry hopping, a cake filtration was performed on a laboratory scale. The filter consisted of a stainless steel filter vessel with a pressure and temperature sensor and a scale (see Fig. 1a). The sensors and scale were recorded using a computer with the program Virtual Expert (Gimbio GmbH, Freising, Germany) [23]. Filtrate volume (g) in dependence of filtration time was recorded and subsequently flux through the hop filter cake was calculated. The filtrations were carried out at room temperature. At the beginning, 500 mL of sample with 5 g/L hops were filled into the filter vessel and filtered (see Fig. 1b—dashed line). However, there were large deviations in filterability. For this reason, initially 50 mL of distilled water was added in the filter and then the sample (500 mL) was filled in. This experimental setup showed acceptable reproducibility (see Fig. 1b—straight line). All experiments were carried out in triplicate. The samples were filtered through a steel sieve with a pore size of 15 µm at a constant pressure (1 bar). The hop particles retained by the filter medium build a filter cake, which has a higher flow resistance with increasing time. For comparison of the hop products, flux through the hop cake in $g/(cm^2 \times min \times bar)$ was calculated.

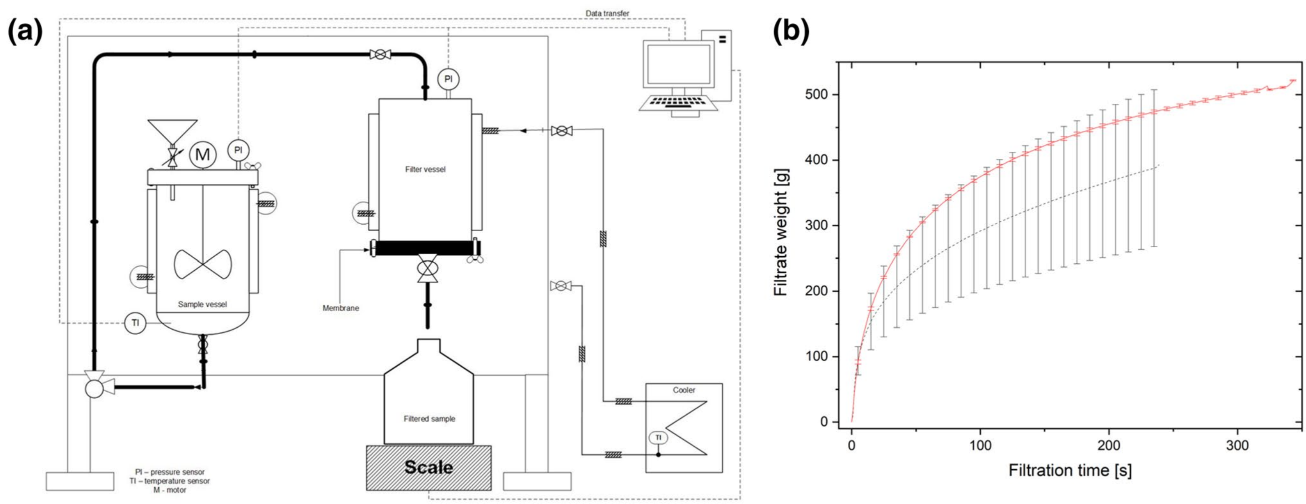

Fig. 1 The filter equipment (**a**) used for the filtration trials. The filter consisted of two stainless steel vessels with a cooling jacket which are connected to a cooler. Pressure (PI) and temperature sensor (TI) as well as a scale were connected via a controller to a computer (data transfer—dashed line). The entire setup is mounted on a steel frame and connected with hoses [23]. Examples of hop filtration trials (**b**) (H HEB, $n = 3$) without a template of distilled water (dashed line) and with a template of water (straight line). Error bars represent the standard deviation

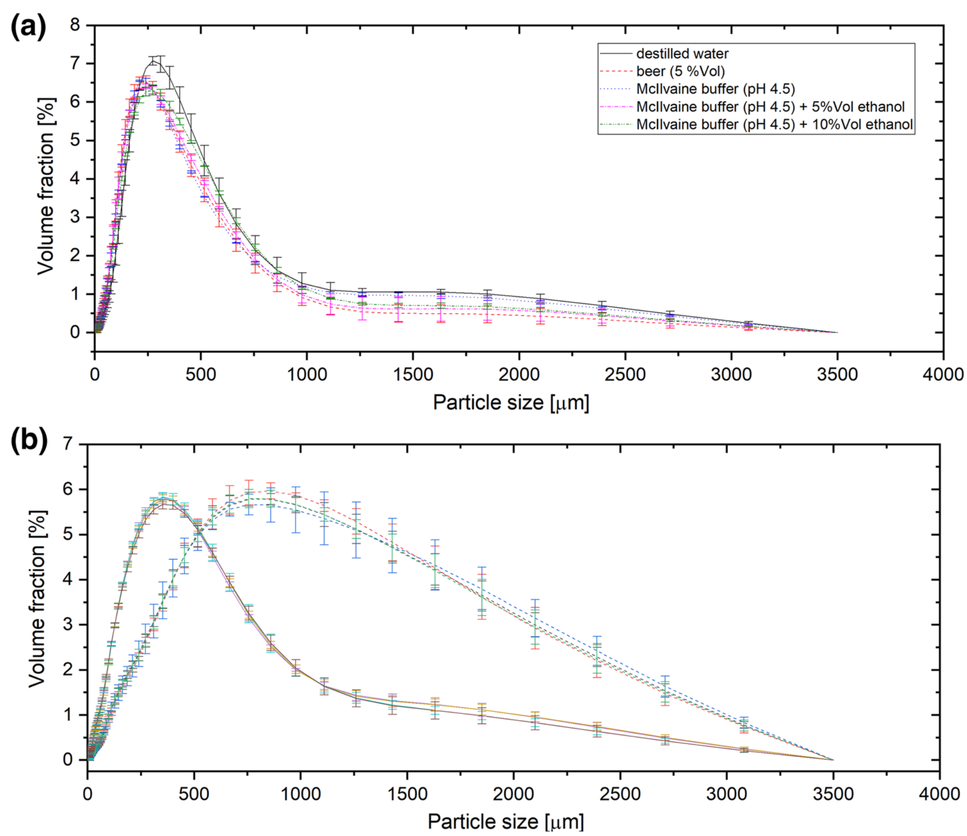

Fig. 2 Impact of the solvent (**a**) (distilled water (compact line), beer (5 vol%—dashed line), McIlvaine buffer (pH 4.5—dotted line), McIlvaine buffer with 5 vol% ethanol (pH 4.5—dashed–dotted line) and McIlvaine buffer with 10 vol% ethanol (pH 4.5—dashed–dotted–dotted line) on particle size distribution ($n = 20$) of dissolved hop product (H PLA) and repeatability (**b**) ($n = 5 \times 5$) type 45 (compact line) and type 90 (dashed line) pellets H HTR in McIlvaine buffer with 5 vol% ethanol

Statistics

The statistical analysis was carried out with OriginPro 2016G (OriginLab Corporation, Northampton, USA).

Results and discussion

Before the influence of the hop particle size distribution on its filter performance could be investigated, a reproducible methodology for the analysis of particle size considering disintegration and swelling was needed. Furthermore, additional information on the hop pelleting process and its impact on the particle size should be examined with this method.

Impact of solvent on particle size distribution and repeatability

To analyze the particle size of the hop constituents, various influencing factors had to be examined. At first, hop particles must be disintegrated from the pellets without damage. The solvent should be comparable to results obtained in beer. For this reason, distilled water, citric acid phosphate buffer and citric acid phosphate buffer with 5 and 10 vol% ethanol were tested and compared to the hop particle size in decarbonized lager beer (5 vol%). Thus, particle size distribution of Polaris (H PLA) type 90 pellets was measured in these different solvents after a comparable solving time of 60 min (see Fig. 2a). In addition, this experiment was repeated with the variety Hersbrucker (H HEB, data not shown) and Hallertauer Tradition (H HTR, data not shown). It could be shown that the particle size distributions in all tested solvents were comparable and occurring deviations lay within the measuring accuracy. Nevertheless, the best comparability to beer was observed in buffer with 5 vol% ethanol. This could be confirmed in two further hop samples (flavor and bitter hops).

Repeatability of particle size measurement was tested in two different hop pellet types (45 and 90), shown in Fig. 2b. It could be shown that type 45 pellets had a smaller particle size distribution than type 90 pellets of the same hop variety (H HTR). The deviation of the five measurement approaches was very small, which indicates a reproducible sample preparation and measurement. To test the repeatability of the method, eight further hop samples were examined on their average particle size *Dx*(50) (see Fig. 3). The repeatability of the method could be determined with an average standard deviation of 25.9 µm and a confidence interval of 3.78% ($n=180$). A larger average particle size resulted in a higher deviation (see Fig. 3—sample H HTR and H HEB). Since a good reproducibility of the method could be determined, the next step had to be the investigation of hop swelling and its impact on particle size distribution.

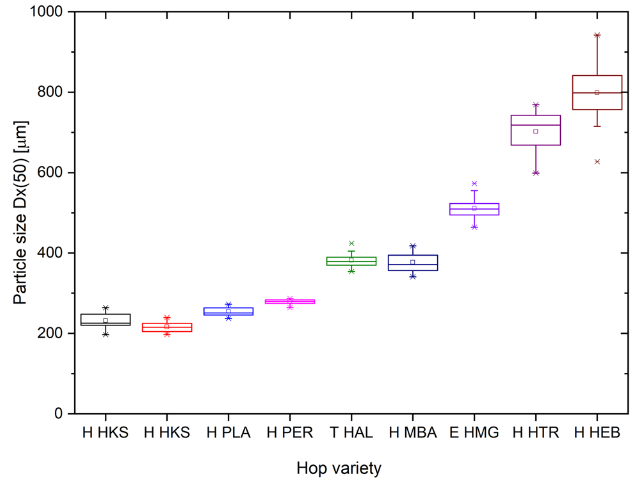

Fig. 3 Repeatability of average particle size *Dx*(50) ($n=20$) of different hop varieties in McIlvaine buffer with 5 vol% ethanol

Influence of the swelling behavior on the particle size distribution

The investigation of the optimal measurement time was necessary to exclude possible changes due to the swelling of the hop constituents on particle size. Thus, the aim was the examination of that time from which the particle size no longer changes due to swelling and a stable measurement was possible. For this reason, the particle size of various hop pellets was first determined within 1 h in a 10-min cycle and then within 3 h in a 30-min cycle.

It was noticeable that the particles swelled up to 30 min (see Fig. 4a). Thereafter, only a small change in particle size could be determined. Between 1 and 3 h, almost no change in particle size could be detected (see Fig. 4b). These experiments were repeated with a hop with lower α-acid content (H HEB type 90 pellets). Comparable results for the swelling time were determined with these (data not shown). It could be observed that the sample with a lower α-acid content had a greater swelling (15% H HEB type 90 pellets) than the sample (10% H HMG) with a higher α-acid content.

In addition, the average particle sizes *Dx*(50) were compared to obtain significant differences using a one factorial ANOVA (Tukey test). The test confirmed significant differences in *Dx*(50) up to 30 min. Thereafter, no significant differences in particle size could be determined. For this reason, all further measurements were performed after a swelling and disintegration time of 1 h.

Impact of pelletizing process on hop particle size

Since a smaller particle size distribution of lupulin-enriched type 45 pellets could be determined, the pelletizing process and its impact on the particle size should be investigated in

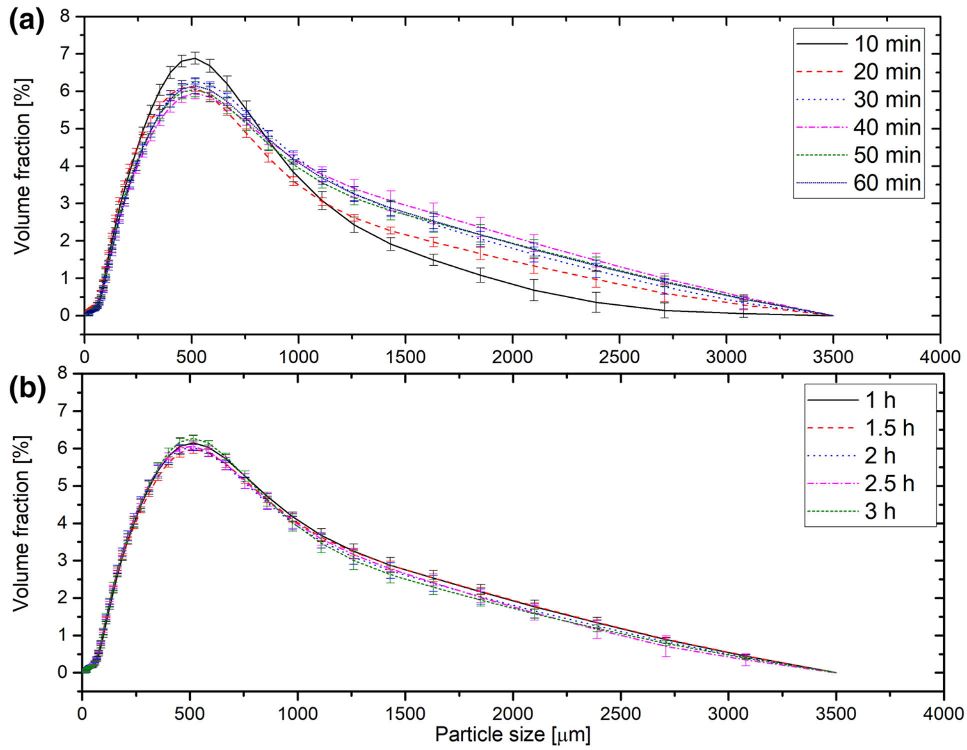

Fig. 4 Influence of time on the swelling of hop particles (H HMG type 90 pellets) shown as particle size distribution ($n = 20$) in McIlvaine buffer with 5 vol% ethanol, 0–60 min in a 10 min interval (**a**), 1–3 h in a 30 min interval (**b**)

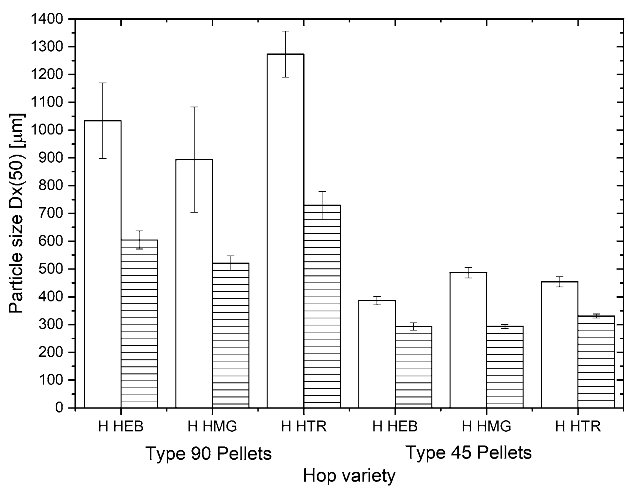

Fig. 5 Impact of pelletizing on average hop particle size (*Dx*(50), $n = 5$) of hop powder (empty bars) and hop pellets (striped bars) for type 90 and type 45 pellets

more detail. That is why the powder before pelletizing and the associated pellets were examined on their particle size distribution. The measurement showed that the hop powder for the conventional type 90 pellets contained more coarse constituents (see Fig. 5) and thus has a larger average particle size. This can be explained by the greater purification due to sieving of the type 45 powder during processing and its associated higher proportion of lupulin. This lupulin has a particle size of about 200 µm and a high content of this ingredient is the main objective in the production of type 45 pellets [1]. The results of the previous measurements that type 45 pellets have a smaller particle size distribution could be confirmed. However, only a slight reduction during pelletizing was determined. The particle size decreased by 24.1 (H HEB), 27.1 (H HTR) and 39.6% (H HMG) (see Fig. 5). In comparison, a higher reduction of the particle size was determined for the type 90 pellets. The hop variety H HTR had the largest particle size reduction of 42.7%. The other hop varieties decreased by 41.5 (H HEB) and 41.7% (H HMG), respectively. The results showed that pelletizing causes a further reduction of the particle size of the hop constituents. Due to the smaller particle size of the raw material (powder) and processing at low temperatures, which is needed for the purification of the sticky lupulin, however, this reduction is smaller in the type 45 pellets.

To what extent this reduction affects the filtration performance of a beer hop mixture is considered in the following chapter.

Impact of the solvent on filterability of hop products

In addition to the particle size measurement, the filtration performance of the hop products should be investigated. As with the particle size measurement, the influence of the solvent on the flow of the hop products should be tested. 5 g/L of hops was weighed and mixed with the

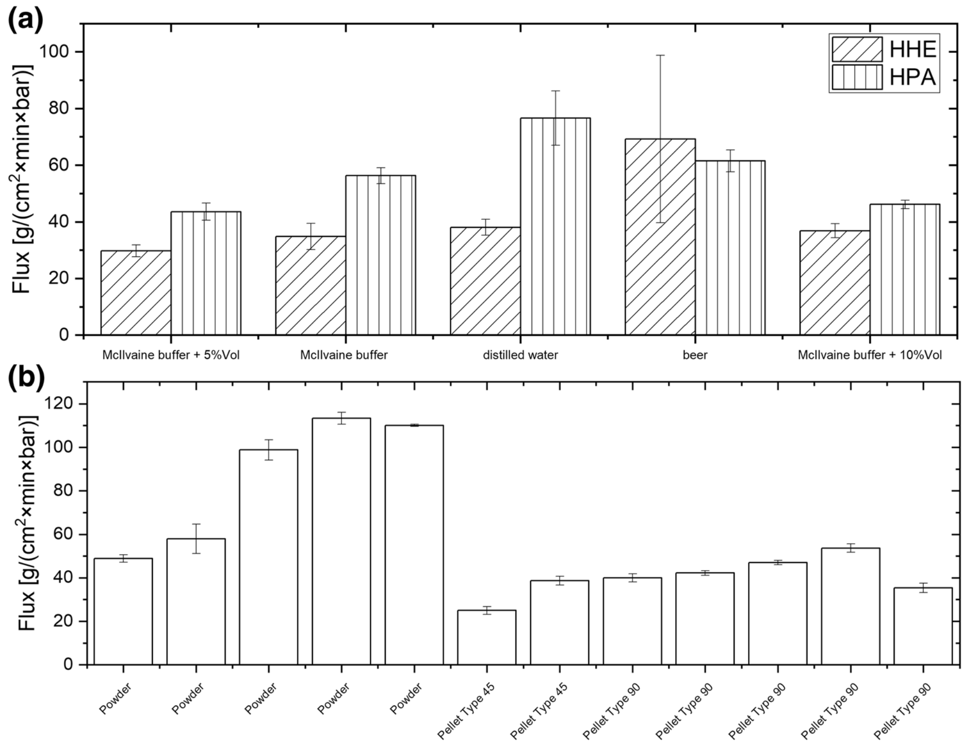

Fig. 6 Impact of the solvent on the flux (**a**) ($n = 3$) of the hop products H HEB and H PLA (both pellets type 90) and comparison of the flow rate (**b**) ($n = 3$) of different hop products and varieties

appropriate solvents. The previously determined swelling time was also applied in these experiments, which is why a contact time of 60 min was chosen. This was necessary to disintegrate the hop pellets. In addition, a swelling of the hops occurs during the dry-hopping process. Figure 6a shows the flux in [g/(cm^2 × min × bar)] of two different hop varieties. McIlvaine buffer (pH 4.5) with 5 vol% ethanol had a comparable filterability to pure buffer (pH 4.5) and buffer (pH 4.5) with 10 vol%. Distilled water was not comparable to buffer or beer. The results of beer were not comparable to the flux of buffer. But since McIlvaine buffer with 5 vol% ethanol had best comparability in particle size distribution to beer and a good reproducibility of the filtration trials, further experiments were performed with this McIlvaine buffer with 5 vol% ethanol.

To investigate the reproducibility of the method, different hop pellets and powders were filtered in laboratory scale. The flux of the hop products ranged between 20 and 120 g/(cm^2 × min × bar) (see Fig. 6b). Hop powders had a higher flux than hop pellets. There were no major differences between the pellet types, but the type 45 pellets had a slightly lower flux. This can be explained by the higher enrichment of lupulin and the associated lower average particle size. The average standard deviation could be examined with 2.35 g/(cm^2 × min × bar) and a confidence interval of 0.77% ($n = 36$).

Since the investigated hop products showed large differences in flux and particle size, a possible correlation should be examined. Furthermore, it should be investigated whether coarse hop constituents have a better filter performance during dry hopping.

Comparison of particle size and filtration trials

To determine the influence of hop particle size on filter performance, 58 hop samples (different varieties) were purchased from all over the world (type 90 and type 45 pellets as well as some powdered hop samples) and analyzed with the methods described above. Most investigated samples had a flux between 25 and 60 g/(cm^2 × min × bar) and an average particle size ($Dx(50)$) smaller than 700 µm). Only five samples had an average particle size above 800 µm. Furthermore, two samples had a flux smaller than 25 g/(cm^2 × min × bar). These samples had not only a low flux, but also a high standard deviation in average particle size. Furthermore, it was found that the particle size of the powdered hop products had a larger standard deviation especially in samples with average particle size above 800 µm. Only one of the investigated pellet samples had a mean particle size greater than 600 µm, which confirms a further reduction of the hop leaf material during the pelletizing process. Again, no major differences between type 45 and type 90 hop pellets could be found. The average flux of type 45 pellets was determined with 41.4 g/(cm^2 × min × bar), whereas type 90 pellets had 45.0 g/(cm^2 × min × bar). 82% of type 90 pellets had a flux above 40 g/(cm^2 × min × bar). In contrast, the type 45 pellets had a share of only 57%. These results are associated with the particle size data. Only 14% of the type 45 pellet had an average particle size above 400 µm, whereas 35% of the type 90 pellets exceeded this size. This shows that type 90 pellets usually have a higher flux, but type 45 pellets, when properly processed, can also produce high flux values.

Nevertheless, a linear correlation between average particle size ($Dx(50)$) and flux could be determined with $r = 0.701$, $P < 0.001$ (all hop products), wherein two outliers (see Fig. 7—samples A and B) could be identified. To check the particle size measurement of these samples, a microscopic examination was performed.

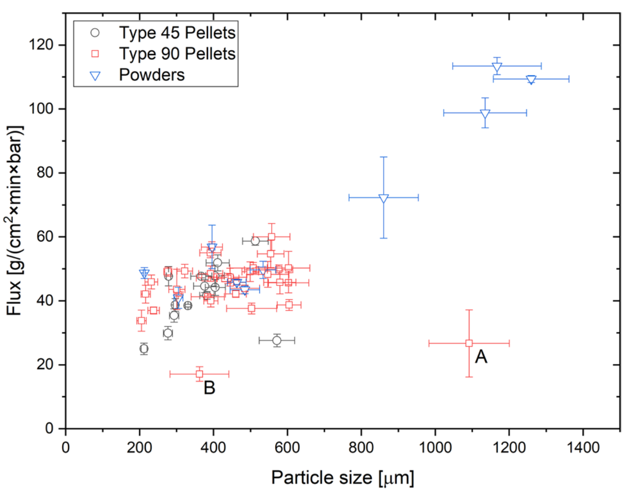

Fig. 7 Comparison of average particle size ($Dx(50)$) ($n = 20$) and flux ($n = 3$) of 58 different hop products

According to the particle size measurement with Malvern Mastersizer, the average particle size of hop product A was determined with 1092 µm and 362 µm of hop product B. Microscopic analysis, shown in Fig. 8, observed very big particles greater than 3500 µm in each hop product. Since maximum measureable particle size of the Malvern Mastersizer was 3500 µm, these particles were not included in the calculation of average particle size. Furthermore, such a hop particle distribution appears to have a negative effect on filter performance due to a layering of big particles in the compressible hop filter cake.

Without these two outliers, a significant correlation ($r = 0.846$, $P < 0.001$) between flux and average particle size of the hop products could be determined. Thus, the coarser the particle size of the hop product, the higher is the flux, and therefore the better is the filterability. However, this phenomenon seems to turn negative for hop products containing particles larger than 4000 µm (big hop leaves, see Fig. 8a) as well as very small particles (see Fig. 8b). Thus, these measurement results provide new insights into the behavior of hop products during filtration using special dry-hopping equipment and thus provide the basis for optimizing the production of hop products and especially hop pellets.

Discussion

The use of hops in dry hopping has different requirements than in the brewhouse. But currently, there are no differences between the hop products for these two applications. This is not at least the case, since a lack of information about the particle sizes of hops and their influence on the filter performance. However, initial findings of the literature show that measurement methods for the determination of particle

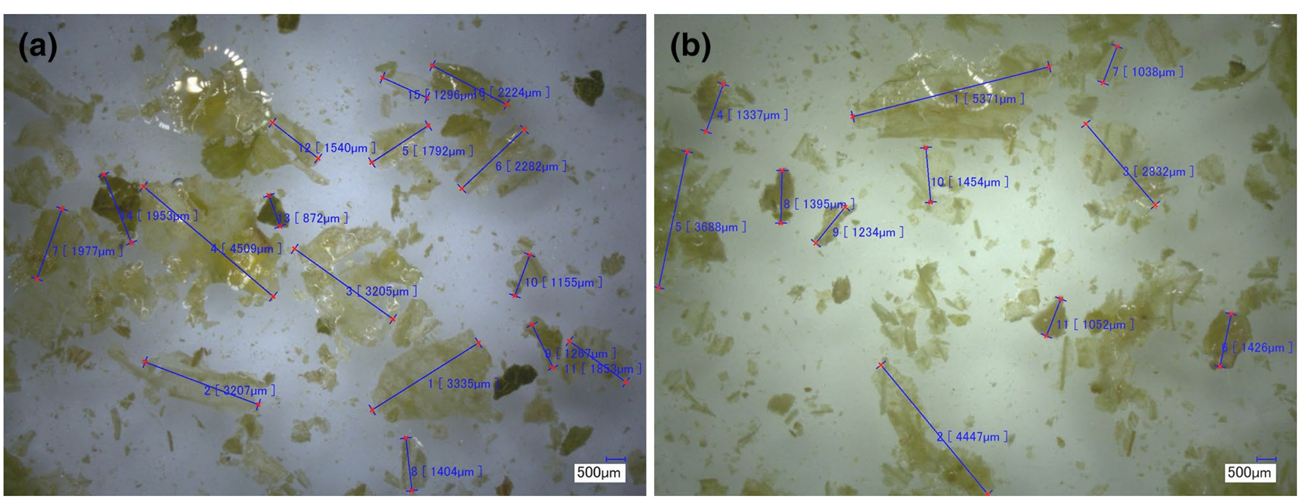

Fig. 8 Microscopic analysis of the two hop products with the lowest flow rate (outliers)

size and filter performance could be useful to optimize the processability and yield during dry hopping.

According to the literature, various influencing factors on the disintegration and swelling of hop pellets could be observed. Engstle et al. [24] examined that the α-acid content of the hops has an influence on the swelling of the pellets. They were able to show that pellets with a high α-acid content had a slower disintegration due to the hydrophobic nature of humulones and the lubricating effect of hop resins. This could also be confirmed with the data presented here. A swelling time of 2 h with distilled water as solvent was used by the authors [24, 25], which corresponds to a doubling of the analysis results presented here. However, the investigation on swelling time showed no significant change after 30 min. In addition, no experiments on swelling and disintegration of pellets in beer were performed by the authors [24, 25], which does not allow any information on the extent of water as a suitable medium for detecting the disintegration of hop pellets during the brewing process. However, the results of the solvent comparison show clear differences between water and beer, which was shown by the studies presented above. In addition to swelling and disintegration, the particle size distribution of five hop varieties was measured by laser diffraction and quasi static image analysis, wherein no information on measurement accuracy and maximum measurable particle size was shown by the authors [24].

In comparison, Wolfe et al. [13] used sodium citrate buffer (pH 4.2) with 6 vol% ethanol to determine the effect of hop particle size on the flavor intensity. Although the investigated pellets showed significant differences in particle size, a maximum time for disintegration of 30 min was determined for all pellets. These findings are comparable to the previously shown data (see Fig. 4). The particle size measurement was performed with the help of five sieves (2.36, 1.20, 0.59, 0.25, and 0.15 mm) and disintegration was carried out in water (20 °C) with subsequent drying of the particles (overnight) [26]. Thus, the results hardly allow a conclusion on the distribution of the hop particles in the pellets and, additionally, this method includes potential sources of measurement errors, such as the agglomeration of hop particles. In addition, this measurement does not involve swelling of the hop particles (due to overnight drying), which does not provide accurate information for the later dry-hopping process. However, since all investigated pellets are disintegrated within 30 min, it can be assumed that the disintegration can be neglected for the dry-hopping processes, since the typical timescales are 24 h to 1 week [13, 20].

Additionally, the impact of particle size on hop flavor in beer was investigated by different authors. Due to the high volatility of some hop flavor substances (e.g., myrcene), they evaporate during wort boiling and therefore have little impact on the beer flavor. However, these substances can be enriched in beer during dry hopping, resulting in a respective typical hoppy and fruity aroma. Different authors have stated that hop particle size may have an impact on distribution in storage tanks and thus the aroma-entraining substances in beer during dry hopping [13, 27, 28]. These aroma substances and hop oils are contained in the lupulin glands. The availability of hop aroma is impacted by integrity of the lupulin glands, since an intact membrane aggravate extraction of aroma substances. During pelletizing, lupulin glands are squeezed and thus hop oil is easily available [29]. Investigating four different hop pellets, Wolfe et al. [13] could not find significant differences in linalool and myrcene concentrations in beer on days 1, 4 and 7 of their dry-hopping trials. In addition, extraction of the flavoring substances was examined within the first 24 h after addition of the hop pellets and whole hop cones. These experiments showed differences in the terpene alcohols, which were extracted very quickly at first and then either remained static or increased very slowly, and the hydrocarbon compounds, which were fully extracted in as little as 4 h followed by a decline. Schönberger et al. [27] investigated four different pellet types (type 90, purified type 90, type 67 and type 33) of the variety Hallertauer Mittelfrüh on the impact of hop aroma in beer. Since the content of lupulin and thus the oil concentration of the pellets varied widely, the concentration of the products during dry hopping was adjusted to a constant hop oil content. The produced beer samples had not only clear difference in sensorial evaluation, but also the content of flavoring substances such as myrcene and linalool varied significantly. This shows that the depletion of leaf material in the pellets can result in a change in hop aroma, especially a decrease in vegetable aroma substances. Further information on the particle size of the products and the dry-hopping process cannot be found in the publication [27].

The presented literature shows that there have been no systematic investigations of hop particle size and the applied dry-hopping method on beer flavor, not the least because the particle size measurement of hop pellets is a complicated process. Nevertheless, first information on the sedimentation of hops in the storage tank depending on the particle size can be found in the literature. Thus, for a better extraction of aroma compounds, hop should sediment slowly, which can be observed especially with fine hop particles [13, 28].

The presented data show that a reproducible method for particle size measurement of hops could be set up (see Figs. 2b, 3), which allows not only the optimization of the dry-hopping process, but also the pelletizing of the hops. In addition to the determination of a practical solvent for hop swelling, an optimal disintegration time of the pellets could be examined. Thus, the application of the method allows the investigation of all processing steps during pelletizing and, consequently, optimizations in the particle size distribution of the hop constituents. Furthermore, it could be shown that not only the crushing of the hop constituents during processing, but also the pelletizing itself has an impact on the particle size distribution in the pellets.

In addition, the investigated solvents did not only affect the particle size distribution, but also the filtration performance of the hops due to their different ability to solve hops ingredients. Using McIlvaine buffer (pH 4.5 with 5 vol% ethanol), a reproducible method for the characterization of hop powders and pellets for the investigation of filtration performance could be determined. Nearly no information between the optimal particle size of the hop and filter performance of external dosing systems can be found in literature. Only Bandelt Riess et al. [25] described a method for the determination of permeability of hop particles. The authors used 20 g hops in 1.2 L water at a filtration pressure of 0.2 bar. In addition to the experiments with water, beer was filtered with varying concentrations of yeast. It was found that the permeability decreases with increase in cell count. In addition, the authors described that the porosity of the filter cake was reduced by small particles [25]. This confirms the presented data that hop products with a small particle size have a lower flux (see Fig. 7). However, cake formation is not only influenced by the size, but also the shape of the particles. The presented results suggest that coarse hop constituents increase filter performance. However, it could be examined that large hop leaves can also impact the filtration, because they cannot form a permeable cake due to layering. To carry out a characterization of the hop with regard to its filtration properties during dry hoping, the use of both analytical methods (particle size and flow measurement) is essential. The results presented offer optimization options in terms of hop pellet processing, such as crushing, sieving and pelletizing. It was found that powders had a higher filter performance. In addition, the sieves of the hop filter systems can be adapted to the hop products used to increase the filter performance. To what extent the hop particle size in these systems affects the aroma must be the subject of further investigation.

Conclusion and outlook

The hop pelletizing process is mainly based on empirical knowledge about the use of different sieve sizes during milling, depth of the dies and maximum application temperatures. In this case, it is not so much the field of application for which the pellets are produced, but above all the α-acid content of the hops which decides the way of processing. An objective can be to receive stable pellets with low amounts of hop dust, which is helpful for these brewers using automated dosing systems. A stable pellet is connected to a higher pellet temperature during pelletizing, but higher temperatures could result in an increase in hop storage index (HSI) as well as a decrease or change in the oil content [14]. For this reason, it is important for the pellet manufacturer to find a balance between the physical integrity of the pellet and the hop quality.

The shown method allows a comparable particle size measurement of hop pellets and powders. Using the solvent citric acid phosphate buffer (pH 4.5 + 5 vol% ethanol), a comparable particle size distribution as in beer could be achieved, which suggests a similar swelling behavior. Thus, a reproducible test was developed to investigate hop products and their influence on filter performance to separate hops after dry hopping. Furthermore, an optimal time for disintegration and swelling of 1 h at room temperature in buffer (pH 4.5) could be determined. Compared to the sieving method, the use of the Mastersizer with hydro dispersion unit allows the determination of the hop product particle size distribution in a homogenous suspension. Thus, the described method permits the detection of the hop particle sizes under trial conditions close to the brewery. For this reason, this method can be used as a tool when navigating hop suppliers or working with hop suppliers to optimizing filtration performance using particle size control during pellet production.

The developed methods form the basis for the investigation of the particle size of hop products and their effect on the filterability during dry hopping. Further investigations have to show to what extent the particle size distribution of the hop products effects beer flavor and aroma yield. The findings must be taken into account in the processing of hops to optimize the production of dry-hopped beer.

Compliance with ethical standards

Conflict of interest The author(s) declare that they have no competing interests.

Compliance with ethics requirements This article does not contain any studies with human or animal subjects.

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