J. Zacharias, R. Schneid, and R. Scholz

Precoating Filtration with Compressible Filtering Aids featuring Viscose Fibres

Viscose fibres are described as being the key to using alternative filter aids successfully for precoating filtration in beer clarification jobs. The process characterisation of the fibre attributes, in particular with regard to their adjusting permeability in the filter cake, and their adjustable porosity in accordance with the general controlled process variables of the filtration system, are described as the key factors for utilisation of these fibres. As a result, it is shown that the compressibility of this filter aid has to be integrated into the filtration strategy here. This approach is validated with illustrated filtration results, and described on the basis of a development process actually carried out.

Descriptors: beer filtration, precoat filtration, viscose fibres, alternative filter aids, compressible filter media, TFS/twin flow system

1 Introduction

Worldwide, the overwhelming majority of filtered beer is filtered with precoating filtration systems using kieselguhr as the filter aid. Kieselguhr has many advantages, but also many drawbacks, particularly in regard to its disposal [e.g. 1]. This is why breweries, research institutes and the vendors concerned have been seeking out alternatives for some years now.

In a large number of publications [e.g. 2, 3, 4], specialist literature has reported on the history of filtration in general and on the scientific and industrial activities undertaken in order to develop different alternatives. Yet the question as to where the trend will lead in the future remains unanswered. Furthermore, over the last few years various attempts have been made to achieve “pure” types of cellulose in a thrust towards developing more stable alternatives [e.g. 6, 7]. In particular, an account of these attempts is provided, and the methods of resolution discussed in the selection of papers below:

Braun [5, 6] describes the use of cellulose fibres for precoating filtration of beer in his publications. He comes to the conclusion that their use as an alternative aid for filtering beer is possible, but clearly illustrates where the performance limits lie as regards turbidity reduction. They continually oscillate around the upper acceptance limit.

In his paper, *Zellner* [7] investigated the possibilities for developing a filtration procedure featuring the use of regenerative cellulose fibres, while turning his attention to the effect of the cellulose’s compressibility on the progression of pressure drops, permeabilities and filter cake resistances. He came to the conclusion that acceptable beer clarity can only be achieved with tandem filtration concepts (i.e. with two-stage processes). He does not describe a further process strategy.

As regards the general use of cellulose, *Müller* [8, 9] describes the possibility of using blended products (including cellulose) as a component. He also points out that the use of cellulose in the beer industry has been a subject of interest for several years, but that the best filtration results can only be achieved by adding other components.

Alles’ [10] paper investigates process strategies for filtration with compressible filter cakes. Here, no direct reference to beer filtration can be observed. The flexibility of natural fibres is the main reason for the compressibility of the filter cake, not the fibres themselves (as is mostly assumed). She also describes the compressibility as being the porosity subject to the solid matrix pressure and the flow resistance, which also depends on the solid matrix pressure. She discusses the question of the optimum pressure or pressure drop. However, based on one conclusion, she judges the candle filter to be unsuitable for filtration with this medium. With compressible media, a simple increase in pressure is also described as being unhelpful, and as having a mainly negative impact on the filtrate flow. The findings of this work accordingly offered no means of deriving a strategy for beer filtration with compressible fibres, though a basic method of approaching their compressibility is documented.

More importantly, procedural approaches appear to be significant for further development of a process strategy featuring alternative compressible filter aids. Attention should be paid, for example, to skin formation during dead-end filtration [11] indicating that either a thicker skin surface layer or a thinner, pore-blocking, layer is being formed, which can lead to sudden clogging of the filter cake during precoating filtration. In addition to these, *Drost* and *Windhab* [12] also describe the observation of beer filterability as something that should not be ignored. Here, a non-Newtonian flow behaviour while passing through porous media (such as the filter cake) is made responsible for the sudden rise in pressure drops.

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These results point to consistently positive approaches regarding the use of cellulose. However, what they all have in common is that they do not simultaneously solve the technological and economic aspects of filtration. What all of them have in common is that pure cellulose "only" works right at the limit or mostly even slightly above the required clarity of filtered beer. As a conclusion, that means a more stable filtration result is only achievable with some highly disparate additives.

Based on these findings, the authors began to research the use of viscose fibres for the clarification of beer. The aim of the research was to find an alternative filter aid that can be adapted to the beer's filterability and that can overcome the afore-mentioned previous limitations.

With this paper, in a first step, the authors are aiming to demonstrate that there is a way to use compressible filter aids so as to render them beneficial to beer filtration. In principle, this is subsequently described in two main sections, the first of them dealing with the viscose fibres used, and the second covering control of the process sequence and the compressible effects of the self-shaping filter cake.

2 Materials and methods

2.1 Materials

2.1.1 Viscose fibres

A viscose fibre is a man-made fibre composed of regenerated cellulose. Its basis is 100 % cellulose. In general, Viscose fibres are manufactured from pulp in a multi-stage process. The natural cellulose is derivatised in a chemical process to form a solution that is suitable for a wet-spinning process. This solution is pressed through spinnerets into a spinning bath, in which filaments of 100 % regenerated cellulose are formed. The filaments are then stretched, washed, cut and dried in subsequent processes to form the final viscose fibres. Basically, the viscose process simply converts the cellulose of pulp into regenerated cellulose with a custom-made fibre shape. With this process, it is possible to influence the fundamental characteristics of the fibres, which is not the case with the raw materials. Examples of these fibres are shown in figure 1. They are described in detail by *Wimmer* in [13, 14], for example.

The structure and characteristics of viscose fibres can be varied greatly, in particular their length, fineness, shape of the cross-section and surface structure can be influenced. This results in major potentials for creating different filter cake structures that allow for consistent clarification of the beer even if there is a change in the quality of the unfiltered beer. The research project included both the development of suitable viscose fibres and the further development of the filtration process itself.

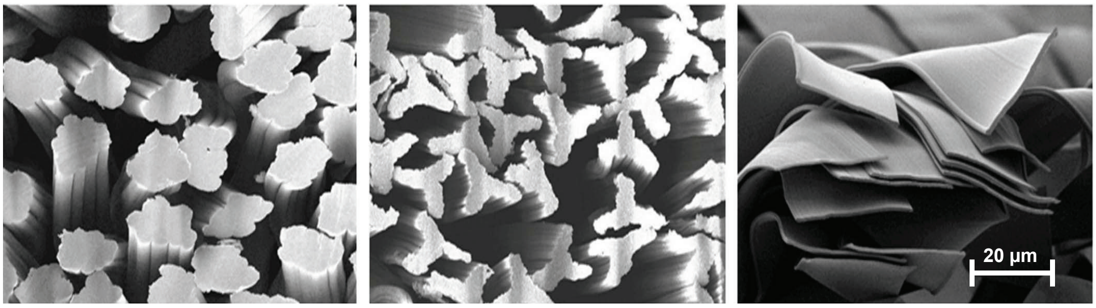

Fig. 1 Scanning electron microscope images of viscose fibres with different cross-sections ranging from 3.3 dtex (Danufil®, Galaxy®) to 9.0 dtex (Leonardo®)

Table 1 Viscose fibres used for the filtration studies presented

Fibre designation	Characteristics
Danufil®	3.3 dtex, used with 50 and 300 µm (q_0)
Galaxy®	3.3 dtex, used with 50 and 300 µm (q_0)
Leonardo®	9.0 dtex, used with 50 and 300 µm (q_0)
GA30	Specific Galaxy® blend with a nominal average length of 30 µm (q_0) and 3.3 dtex

As subsequently described, the formation and structure of the filter cake during precoating is essentially governed by the geometry of the fibres, meaning the shape of the fibre cross-section, its fineness and length. It is expected that the filtration result will be closely dependent on the characteristics of the fibres concerned.

A further major advantage of viscose fibres is that they are based on the sustainable natural polymer cellulose, and that they are completely biodegradable. They are not classified as health-hazardous [15]. They simply need to be classified in accordance with their particle size distribution under the corresponding particulate matter categories in the protective measures and work regulations, and to meet the usual workplace regulations covering dust inflammability. Since the particle size distribution of fibres in their dry condition is between 20 and 300 µm, they are viewed as being an inhalable fraction of total dust, which does not pose a risk to health [16]. The potential risk is comparable to that of pure cellulose.

In this paper, the fibres listed in table 1 were used. They are all produced by Kelheim Fibres GmbH. The designations are registered trademarks. The two main characteristics are fibre length and dtex. The length is the nominal mean value of the fibres' particle distribution, the number average (q_0). The length-related dimension dtex is customarily used in the fibre industry instead of the diameter. dtex is defined as 1 dtex = 1 g per 10^4 m fibres.

For use as a precoating filter aid or the further body feed no additional preconditioning of the fibres is necessary. They just need to be dispersed in the dosage vessel.

2.1.2 Reference beer

A reproducible reference beer is also essential for the permeability and compressibility experiments. Here, a reference fluid was designed for this purpose. The recipe for this medium was adapted to make it comparable to the fundamental ingredients of an average Pilsner beer, based on the fundamental technological

Fig. 2 **Filtration cell (left), pilot filter with candles (right)**

characteristics involved: original extract 13.1 %, real extract 5.2 % (w/w), viscosity 1.7 mPas, colour 9.2 EBC, turbidity 90°/25° at 20 °C with > 99 EBC.

2.2 Methods

Generally speaking, there are different filter systems suitable for precoating filtration; these, are also described in reputable brewing literature [e.g. 17]. For this paper, however, the focus is placed on the options offered by candle filters, and the system is optimised from a process-technological viewpoint. The basics of the candle filter system used (TFS = Twin Flow System) have already been described by [18]. Comparable systems also provide the basis for existing profitability calculations [19] and basic descriptions of procedural backgrounds [20, 10] as well as the suitability of equations using permeabilities, compressibilities, porosities for describing filter resistances, filter procedures and flow rates through filter cakes.

In order to derive any benefit from these foundations, two fundamental steps were performed for the development process. A) The technical centre trial on a horizontal filter frit and B) the pilot trial with filter elements of a TFS pilot filter for beer filtration (see also Figure 2). The technical centre trial characterises the fibre types based on their permeability and compressibility, with due regard to their suitability for the upscaled trial (pilot trial).

2.2.1 Methods for analysing the characteristics of fibres in lab scale

For the basic step, the most important parameters have to be investigated. On the one hand, there is observation of the permeability with its non-dimensional characteristic, which is defined as follows (see Equation 1):

$$ß = \frac{\eta_L \cdot H \cdot V_L}{A \cdot \Delta p \cdot t} \qquad \text{(Eq. 1)}$$

Here, the value ß (often simply also referred to as Darcy) is proportional to the dynamic viscosity η_L of the medium to be filtered and to the height H of the filter cake. In addition, the equation describes the cross-sectional area A of the material through which the liquid flows, the resulting loss of pressure Δp, and the necessary flow time t for a defined sample volume V_L [21]. In the analysis presented, the pressure loss has been set to 0.08 bar, while the temperature of the test media was reduced to 5 °C.

There are different ways of determining the permeability: however, this method is particularly well suited for calculating the permeability of the different viscose filter cakes. It was determined using a simplified outflow and flow-through experiment through a filter frit under defined conditions, as also described by Braun for example [6].

Furthermore, the most common descriptions for the compressibility of a filter cake, the fibres and their determination can be defined using equation 2:

$$\frac{ß_0}{ß} = \left(\frac{p_S}{p_0}\right)^n \qquad \text{(Eq. 2)}$$

This equation results from the mathematical description of filter resistance as a function of solid compression p_S. In this form, the cake resistance is replaced by the permeability ß, meaning the exponent n can be calculated as the compressibility at pressures of p_0 and $ß_0$ [21]. Here, n = 0 indicates incompressible media (such as kieselguhr), values from n > 0 up to n = 1 denote those that are moderately compressible, and values of n > 1 identify highly compressible media. In comparison, the viscose fibres of the trials have values that indicate this moderate compressibility. They are in the range between 0.2 and 0.9, which is why no directly usable information was obtained in regard to the conventional filtration process.

So when it came to fibre observation, this resulted in a simplified test for evaluating flow curves and resulting pressure losses in a reference system, which proved to be a reliable, reproducible tool. It was possible to record this using a simple method in the filtration cell shown (see Figure 2, left) which was equipped with a horizontal filter frit. For analytical purposes, both the volume flow rate through the completely precoated cake and the corresponding pressure loss through the cake were measured using a differential pressure sensor. Therefore, an increasing flow over the precoated frit in the filtration cell (with 150 g/m² body feed) is applied at a media temperature of 12 °C. The pressure drop is measured in dependence on the filter flow.

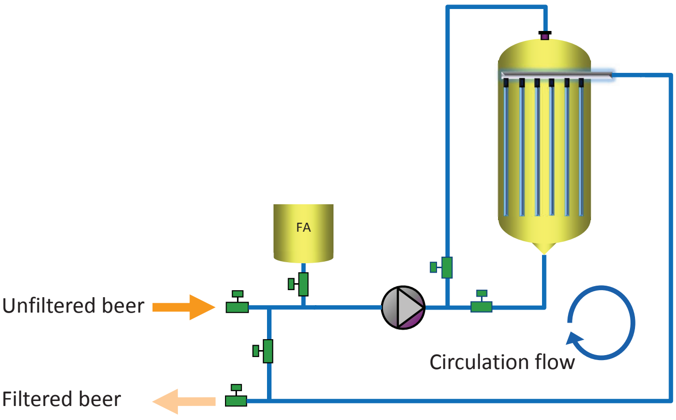

Fig. 3 **Diagram of the TFS filtration and the viscose circulation flow principle**

Nevertheless, porosity ε is defined in accordance with equation 3

$$\varepsilon = \frac{V_{Pores}}{V_{Sediment}} \quad \text{(Eq. 3)}$$

The volume of the pores V_{Pores} is determined by using a cylindrical beaker, subtracting the difference between the practical observed volume and the theoretical volume of the sediment $V_{Sediment}$.

2.2.2 *Upscaling on the pilot scale*

The characteristics (permeability, compressibility, porosity) described in section 2.2.1 created the basis for further optimisation. That subsequently led to upscaling for actual brewery operations, which can be achieved through specific adjustment of the parameters for the viscose fibres, method and process control, in order to enable beer to be filtered there with the requisite quality.

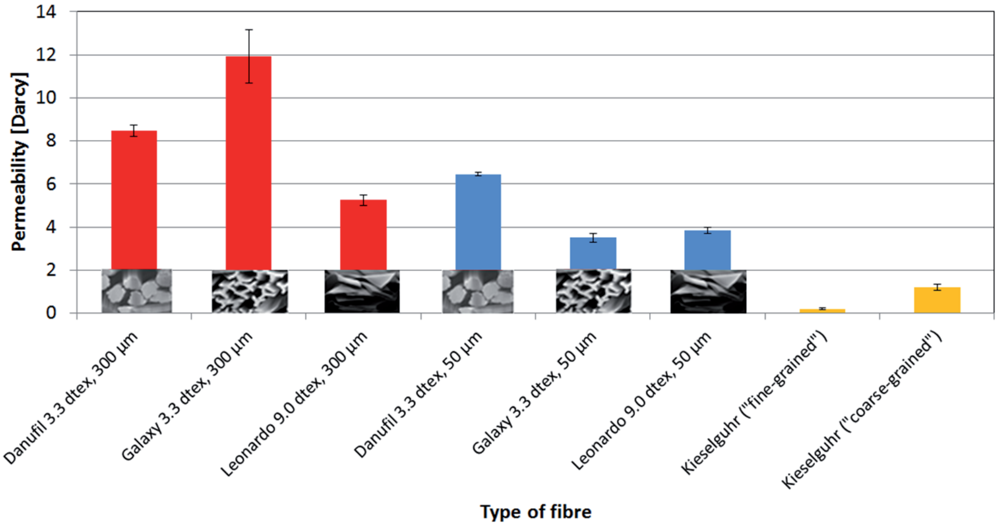

Fig. 4 **Comparing the permeabilities of the viscose fibre types investigated in two different length classes and referenced kieselguhr (level of significance 5 %) at 5 °C under a pressure loss of 0.08 bar**

To ascertain this, the pilot filtration set-up shown on the right of figure 2 was used. Here 7 original Krones TFS filter candles are installed (55-µm gap) in a pilot-scale TFS filtration set-up. Although Alles [10] did not consider it appropriate, this system was in operation for the study presented, because of the advantages expected from the option for implementing different flow directions. In particular, the configuration displayed in figure 3 explains the circulation principle used for viscose cake compression. It is characterised by an unfiltrate inlet that leads across the filtration vessel through the candles to the filtration outlet. In addition, a bypass flow from the filtrate side back to the unfiltrate side is possible. With this flow path, a circulation flow from the unfiltrate side to the filtrate side through the candles is implemented with the TFS filtration system. This circulation flow was used to adjust the necessary compression of the filter cake, which was achieved at a ratio between filtrate flow and circulation flow of 1 : 2 up to 1 : 15, depending on the unfiltrate quality. Reaching this ratio is the starting point for filtration. In the study reported, the initial ratio is 1:10, ending with 1 : 3. To reproduce industrial conditions, the filtrate flow was set to a typical comparable industrial flow (4.5 to 6.5 hl/m²h).

In this pilot-scale set-up original unfiltered beers (type pilsner, “hell” and dark) were used for the filtration trials. For this paper, the results of a type “hell” beer were used at a temperature of 0 °C.

2.2.3 *Laboratory methods*

The analytical evaluations of relevance to brewing technology have been performed by the laboratory of the Technical University of Berlin. The methods used conform to the MEBAK guidelines [22, 23]. BAX/ESR measurement is carried out in accordance with MEBAK, the Central European Brewing Technology Analytics Commission (wort, beer and beer-based beverages), method 2.15.3.

3 Results and discussion

3.1 Results of the lab-scale trials

The results of the permeability and compressibility trials represent the most important foundation for the processing procedure involved in upscaling to the pilot plant.

The permeabilities of different fibres display noticeable fluctuations (see Figure 4) in interdependencies with their different fibre lengths. Here, fibres reach values of between 3.5 and 12 Darcy. In contrast, kieselguhr values that comply with those in the literature [e.g. 6] of between 0.2 and 1.2 Darcy are determined in dependence on their grain size, regardless of the flow rate. When consistent flow rates were achieved, considerable differences become apparent for different fibre types and different length classes.

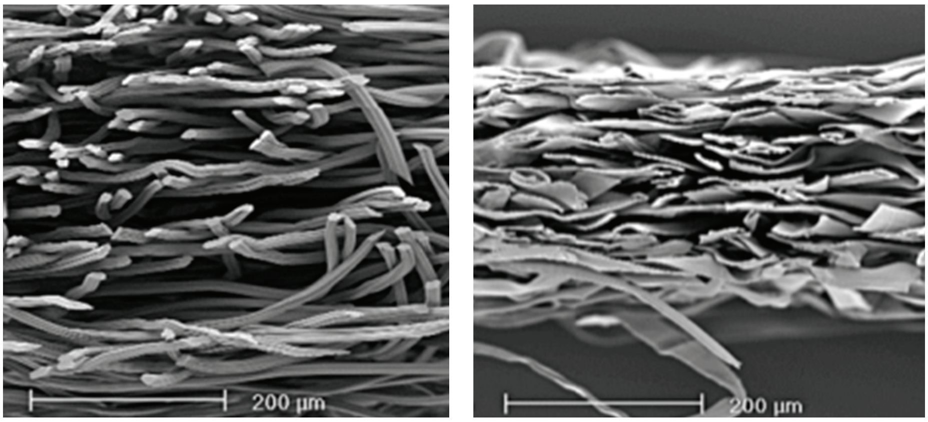

Fig. 5 **Filter cake of flat viscose fibres (e.g. Leonardo®) at two different stages of compression**

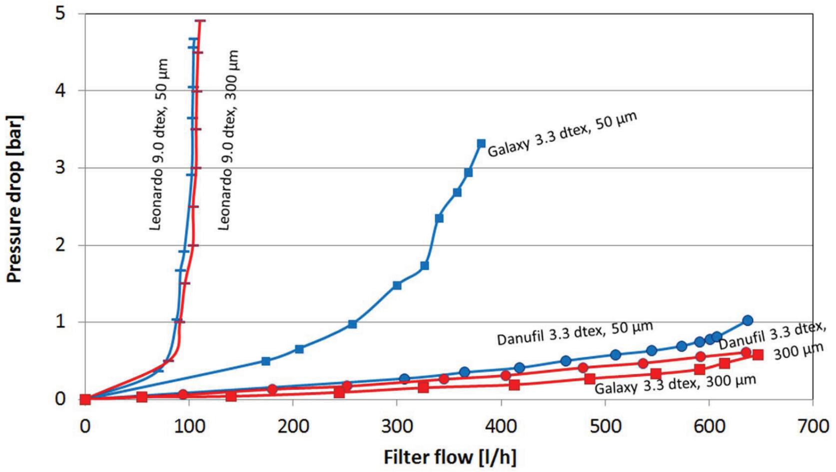

Fig. 6 **Compression developments of selected viscose fibres at 12 °C**

A considerably clearer dependence between different types and size classes is evident here. This is not so recognisable in incompressible filter aids such as kieselguhr. In addition, the dependence of the porosity on the flow speed is manifest. This can be seen most clearly if the cake heights are observed, and their tendency to compression is analysed. The corresponding pressure drops through the cake lead to a clearly visible difference in cake height, as illustrated in figure 5.

If the resulting compression is transferred to measured pressure drops through the cake, an evaluation is possible. Compression and differential pressure developments of the different fibres show a dependence on volume flow rates and the fibre shape, type and size involved. The result presented in figure 6 clearly demonstrates from the compression development that Leonardo® is obviously a fast "closing" fibre, whereas Danufil® and Galaxy® 300 µm tend to be open fibre types, but that there can also be a change to a less fast-closing fibre (e.g. Galaxy® 50 µm). This also shows a selected dependence on the fibre length, something that is noticeable in the Galaxy®, but does not show up at all in the Leonardo® or Danufil® fibre types.

Further trials show correlations between the turbidity achieved and the compressibility of the cake. Fibres that tend to be more compressible achieve lower turbidities but at a lower flow rate. Fibres that tend to be less compressible achieve higher turbidities at a higher flow rate.

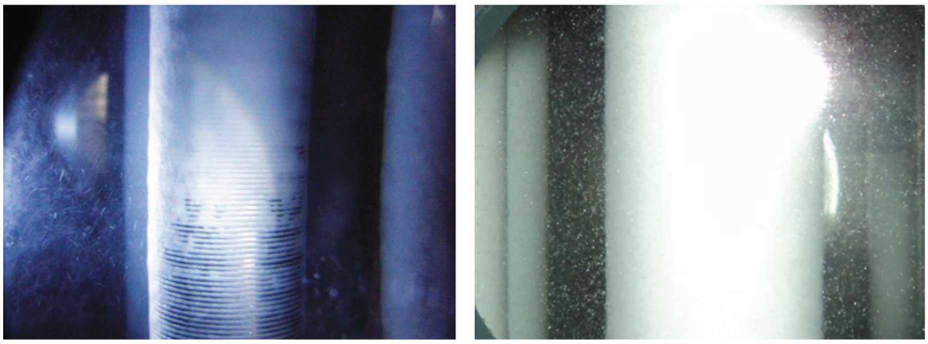

Fig. 7 **Phases for precoating on the candle filter**

One unequivocal result was that single pure fibres did not reach the necessary turbidity levels. It was stated that this can be achieved through special ground and classified fibres or through suitable blends of different fibre types. These findings led to a recognition in section 3.2. that the specific GA30 blend was mixed together for actual use.

As an interim conclusion, it can be stated that the permeability, and hence the porosity, do not lead directly to useful correlations. However, the compressibility of the filter cake can be influenced and adjusted, and is therefore an essential part of the filtration strategy. The shape factor and specific length are important for the build-up of pressure. Due to its composition, the porosity of a suitable fibre blend/distribution must be adjusted using the filtration strategy to an extent that allows values comparable to those achieved with kieselguhr filtration to be reached.

3.2 Results of the pilot-scale trials

Initial observations in the pilot system led to the process of precoating. It performs the task of creating suitable basic precoating layers and validating principles for the test under realistic conditions. Figure 7 shows two different precoating phases. The left section illustrates a formation of a filter cake growing from top to bottom while the right section shows a symmetrical precoating process, which was achieved by adjusting the circulation flow velocities.

Now that uniform precoating had been solved, the next step was to find a filtration strategy that allows the porosity of the filter aid to be controlled during body feed for the filtration of the beer. Additionally while depending on varying unfiltrate qualities the required turbidity values of the beer need to be achieved (25° or 90° turbidity according to EBC).

As Alles [10] and Braun [6] have already shown, different filtration strategies are conceivable when using cellulose or other compressible media. It is clear to see that trials with the most widely used strategy for controlling the constant flow through the filter cake do not have a successful outcome. By contrast, it is particularly apparent that a targeted regulation of the pressure drop through the filter cake manifestly leads not only to the flow being adjusted, it leads also to the result of the filtration, i.e. the quality of the filtered beer. It is thus clearly apparent that the required turbidity values can be adjusted using suitable pressure and flow control (see Figure 8).

It is here that the consequences of a strategy for process control of the flow and pressure ratios in the filter medium can be seen, one that is oriented towards the turbidity of the filtrate and exercises targeted adjustment at the start of filtration. This is achieved with a corresponding high circulation flow. It is further obvious that when the necessary pressure drop (above 1.0 bar) is reached, turbidity stops increasing. And once this condition has been reached, the turbidity levels fall to the necessary values. While this body feed has started, circulation flow decreases to an acceptable level. In the ongoing process, turbidity levels remain constant (at the turbidity levels achieved 0.1 EBC (25°) and 0.25 EBC (90°)) as well as the filtrate flow (300 l/h resp. 5 hl/m²h), while the circulation flow decreases and can be adapted to this requirement by a control. The pressure drop increases slightly up to a constant value. The industrially sufficient flow rate is reached directly after basic precoating, followed by achieving the required turbidities. At this time, the ratio of filtrate to circulation flow is below 1 : 5, and may be even lower. It reaches an optimum after that with a further largely stable filtration phase.

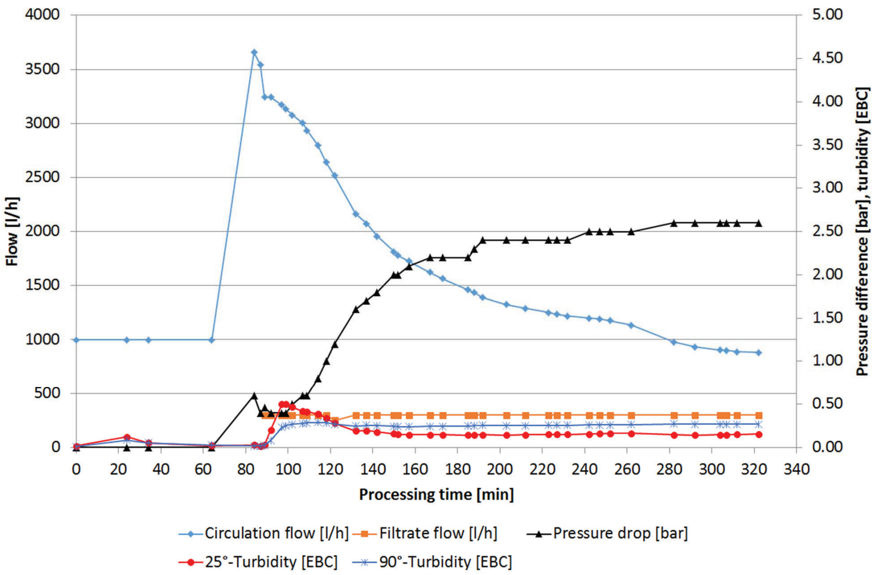

Fig. 8 **Filtration diagram showing an example of filtration with viscose blend GA30; yeast cell count in the unfiltrate is 5.0 million per ml**

During this course of filtration (body feed phase), the filtrate flow tends to be constant, and the development and reaction of the control considers possible changes of the turbidity by adjusting the circulation flow. This means, generally speaking, that the porosity of the cake is regulated using the turbidity values from the filtrate side as control parameters.

In further repeated trial filtrations production times up to ten hours have been achieved with the required turbidities.

The result of the beer quality achieved through filtration using viscose fibres is evidenced through multiple analyses in the laboratory of the Technical University of Berlin (see Table 2 for a complete set of analytical data and Figure 9 for the ESR/BAX evaluation). The beer analyses prove the comparability with conventional kieselguhr filtration. Over all of them, no negative differences were detected between kieselguhr and viscose filtrates. In particular, original extract, colour and head retention show similar values.

Table 2 **Analytical data (by TU Berlin) of the filtration shown in figure 8 ("←*" calculated in comparison to 12 % original extract, because of the pilot plant equipment's slight dilution with the body feed). Filtration routines were performed with identical unfiltered beer from the same fermentation tank.**

Analysis	Unfiltered beer	←*	Kieselguhr filtrates	←*	Viscose filtrates	←*
Original extract [%]	11.70	12	11.35	12	10.77	12
Real extract [% w/w]	4.12	4.23	3.93	4.16	3.75	4.18
Alcohol [% vol.]	4.98	5.11	4.86	5.14	4.59	5.11
Degree of apparent attenuation [% w/w]	80.3	80.3	81.0	81.0	80.7	80.7
Colour [EBC]	7.0	7.2	6.6	7.0	6.6	7.4
pH-value [–]	4.33	4.33	4.33	4.33	4.40	4.40
Viscosity [mPa*s] (12 %)	1.557	1.557	1.537	1.537	1.541	1.541
Bittering units [BU]	18	19	16	17	16	18
Total nitrogen [mg/l] (12 %)	944	944	914	914	916	916
$MgSO_4$ perceptible N [mg/l] (12 %)	219	219	183	183	196	196
FAN [mg/l]	139	143	130	138	128	143
Total polyphenols [mg/l]	264	271	217	229	223	249
Anthocyanogene [mg/l]	70	72	62	66	63	70
β-glucan [mg/l]	245	251	234	249	220	245
Photometric iodine reaction [dE]	0.41	0.42	0.23	0.25	0.24	0.27
Head retention NIBEM 30 [s]	–	–	293	293	301	301
Turbidity 90°/ 25° [EBC] 20 °C	> 99	> 99	0.19 / 0.10	0.20 / 0.11	0.25 / 0.09	0.28/ 0.10
Iron [µg/l]	29	30	75	79	32	36
Copper [µg/l]	67	69	125	130	71	79

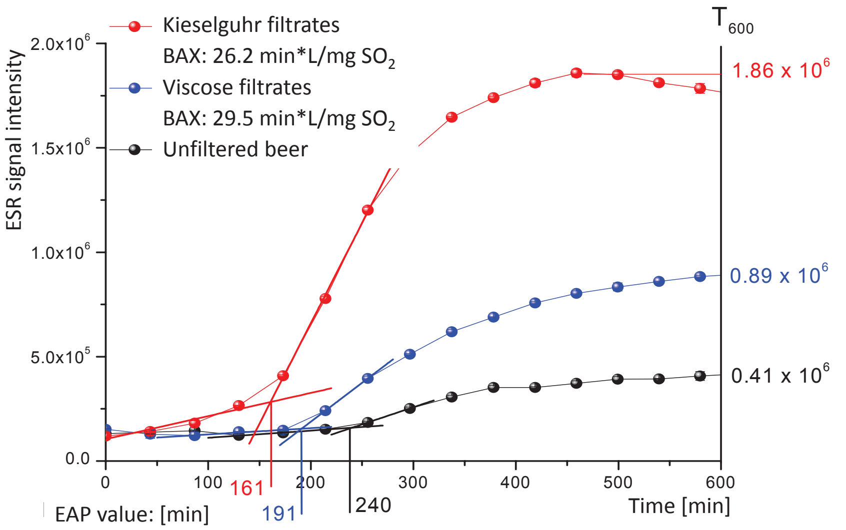

Fig. 9 **ESR measurement of the filtrate using the filtration process depicted in figure 8**

The benchmark of 0.2 and 1.0 EBC is undercut as regards the turbidity values for the significant scattering angles of 25° and 90°. In the viscose filtrate, no yeast cells were found, nor could any other residues such as fibre breakthroughs be found.

Further proof is provided, if you look at the oxidative stability of the filtrate, which is clearly improved due to the minimised iron and copper intake of the viscose fibres. This particularly negative impact (as described in [24] for example) of filter aids such as kieselguhr is therefore omitted. It was clearly observed that iron and copper values, in particular, are significantly lower when using viscose in comparison to kieselguhr as a filter aid. It is also demonstrated by the lower ESR signal intensity as well as the higher BAX values achieved that the viscose filtrate has a much lower oxidative influence on the beer's quality (see Figure 9).

In addition (not reported in this article), the outlook trials of the initial pilot filtration system show that the filtration is stable and can also be adjusted in regard to varying yeast cell counts or beer types, even if further trials still need to be performed here in order to describe this exhaustively. The applicability to further conventional filtration systems also has to be analysed in the next steps.

4 Conclusion

The summarised results of this development project show clearly that with the evaluation of the dependencies of permeability and compressibility of the fibres type, shape and length, a process strategy could indeed be found. This strategy allows acceptable beer qualities to be obtained for filtered beers with viscose fibres. It can thus be clearly stated that, if the existing basics of precoating filtration technology and the results of former applications including pure cellulose fibres are considered, a clear step forward has been made towards achieving an alternative filter aid that is largely suitable for general use. The viscose fibres described demonstrate this. Thanks their purposively shaping qualities, they are the key to achieving stable filtrations during the clarification of unfiltered beer in accordance with the brewery's requirements. Industrially relevant filtrate flows could also be achieved. It is particularly illustrated on the TFS candle filter system used.

It is therefore possible to stipulate that viscose fibres will have a future as an alternative filter aid for precoating filtration as they make a difference in achieving stable process conditions. They are based on the sustainable natural polymer cellulose and are completely biodegradable, do not pose any health risks, and are therefore easy to handle and dispose of; and – what is the most important aspect – they help to achieve an optimum product quality without altering the taste. Functional viscose fibres are a potentially strong alternative to kieselguhr filter aids in the challenging filtration of beer and other foodstuffs. It can be expected that due to this successful adaptation of viscose fibres to suit precoating filtration, breweries, as users of this conventional and globally most widespread filtration technology, will be able to continue production with their existing candle filter lines without having to make major investments in their technology. So precoating filtration with viscose fibres is on its way to becoming a high-performance alternative filter aid.

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