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Filtration of lager beer with microsieves: flux, permeate haze and in-line microscope observations

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Abstract

Membrane fouling during filtration of lager beer with microsieves was studied through in-line microscope observations. It was observed that the main fouling was caused by micrometre-sized particles, presumably aggregated proteins. These particles formed flocks covering parts of the membrane surface. Most of the flocks could be removed by a strong temporary increase in crossflow. Underneath the flocks a permanent fouling layer was formed inside the pores. This made frequent removal of the flocks crucial in delaying the process of permanent in-pore fouling.

Besides the fouling process the influence of pore size on permeate flux and turbidity was investigated. Centrifuged beer appeared to give a significantly clearer permeate than rough beer. For centrifuged beer and a microsieve with a pore diameter of 0.55 μm a haze of 0.23 EBC was obtained during 10.5 h of filtration at an average flux of 2.21×10^3 l/m^2 h. For a sieve with slit-shaped perforations of 0.70 μm × 3.0 μm a haze of 0.46 EBC was obtained during 9 h of filtration at an average flux of 1.43×10^4 l/m^2 h. This flux is more than two-orders of magnitude higher than is commonly obtained with membrane-filtration of lager beer. Concentration of the beer by a factor of 12 hardly influenced the magnitude of the flux. © 2002 Published by Elsevier Science B.V.

Keywords: Microfiltration; Beer; Microsieve; Membrane; Fouling

1. Introduction

Clarification of lager beer is an important operation during the brewing process. Rough beer is filtered in order to eliminate yeast cells and colloidal particles responsible for haze. Common beer-filtration systems are based on kieselguhr. However, the exploitation costs of these systems are rather high. Crossflow microfiltration with polymeric or ceramic membranes may be an alternative. Several studies have been carried out, but often problems like poor permeate quality (i.e. high turbidities or protein and aroma retention) or insufficient fluxes are encountered [1–3]. Moreover, extensive cleaning procedures are required, as beer turns out to cause severe fouling [4–6]. Ceramic membranes have an advantage over polymeric membranes regarding fouling, as they can withstand harsh cleaning methods. However, the obtained fluxes are usually significantly lower. Ceramic membranes with a small flow resistance would, therefore, be highly desirable for beer filtration. Recently developed microsieves made with silicon micromachining are such membranes [7,8]. They consist of

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a thin micro-perforated silicon nitride membrane attached to a macro-perforated silicon support. The membrane thickness is of the order of the pore size, thus, allowing high fluxes and relatively simple cleaning procedures. Moreover, the membrane is optically flat and smooth (surface roughness typically <10 nm), which hampers adsorption of foulants. Furthermore, the pores are uniform in size and distribution, which may be important for the quality control.

Recently, we reported on experiments with yeast-cell filtration of rough lager beer with microsieves [8,9]. Using pore sizes of 0.8–1.5 μm, average fluxes up to 4×10^3 l/m^2 h were obtained. This is approximately one-order of magnitude higher than is commonly obtained for kieselguhr filtration and nearly two-orders higher than for filtration with conventional ceramic membranes. Although the permeate was free of yeast cells, it was still too turbid (0.8–1.2 EBC) for bottling. After several hours of filtration an irreversible fouling layer prevented further filtration. Examination of the sieves showed a remarkable form of fouling. Carpet-like structures covered certain areas of the membrane surface. The origin of these structures was not known, but it was clear that they completely blocked the pores. The SEM micrograph in Fig. 1 shows such a local fouling layer.

Had the carpets slowly grown or were they formed in the system (for instance on the tube walls or in the centrifugal pump) and subsequently dropped on the sieve surface? The best way to investigate the origin of this peculiar form of fouling was in-line observation of the sieve surface through a microscope. We built a set-up for such observations and also investigated the dependence of flux and permeate haze on the pore size. The results give a better insight in the fouling mechanisms and they are very promising for the future application of microsieves for filtration of lager beer.

2. Experimental set-up

2.1. Rig set-up

In order to prevent CO_2 from escaping, beer is normally filtered under pressurised conditions. However, to keep the set-up flexible, we built a rig that can only be used under atmospheric pressure. We realise that the escape of CO_2 will change the pH of the beer and may therewith influence the fouling process.

The rig mainly consists of silicone tubing and was designed for a constant pressure filtration under crossflow circulation of the feed. This constant

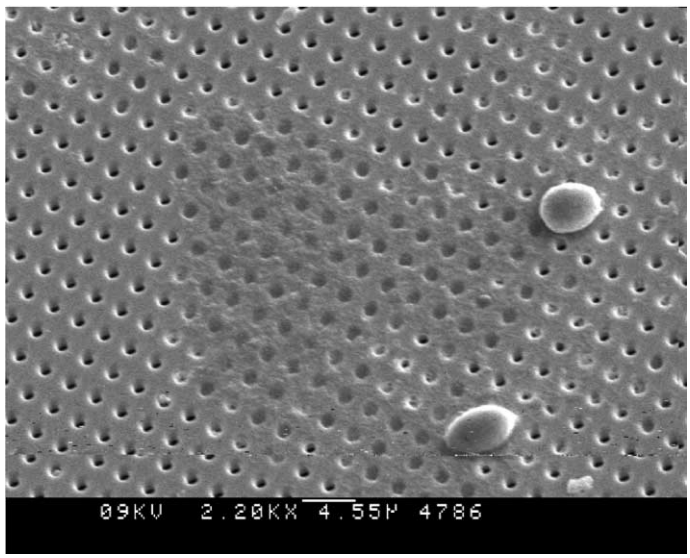

Fig. 1. Carpet-like fouling of a microsieve observed after filtration of rough lager beer.

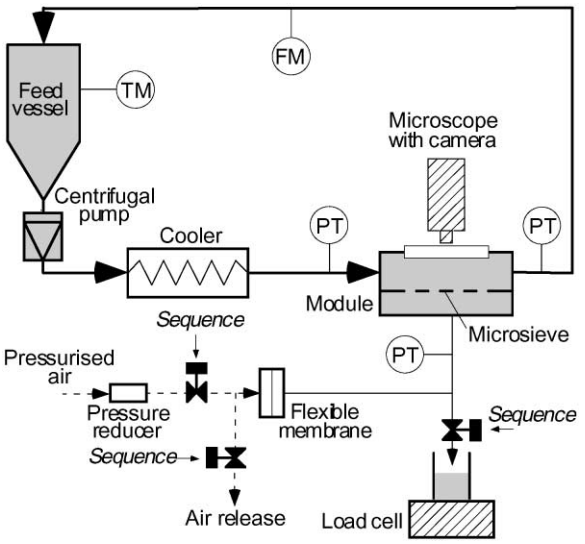

Fig. 2. Schematic illustration of the crossflow rig used for the filtration of lager beer.

pressure is obtained by a difference in height between the feed vessel (volume 2 l) and the permeate outlet. A schematic illustration of the rig is presented in Fig. 2.

Three pressure transducers (PT) are used to monitor the pressure drop over the crossflow channel (ΔP_{chan}) and the transmembrane pressure (ΔP_{mem}). Furthermore, a flow meter (FM) and a thermometer (TM) are used to monitor the crossflow conditions. The upper part of the filter module consists of a glass plate with a 0.17 mm thickness, which allows for observation of the sieve surface through a microscope. The microscope (Leica) is equipped with adjustable objectives, in order to correct for the distance that the light has to travel through the glass and beer. Backpulses are obtained by periodically pressurising the permeate via a dense flexible membrane. In this way no external liquid is added to the permeate. The three valves are actuated all at the same moment with the same signal. The permeate valve and the air-release valve are in a normally open position and the valve in the pressurised-air tube is normally closed. The amount of permeate is measured with a load cell connected to a computer.

2.2. Microsieves

For the experiments several small microsieves were fabricated with an area of 5.5 mm × 5.5 mm. The membranes contain circular pores with diameters of 0.55, 0.80 and 1.5 μm. Fig. 3 shows SEM micrographs of the sieves with 0.55 and 0.80 μm pores.

Furthermore, two membranes with slits were used with a slit length/width ratio of 5. The widths of the slits are 0.70 and 1.5 μm. SEM micrographs of the sieves with slits are shown in Fig. 4.

The membrane thickness is 1.0 μm for the 1.5 μm pores and 0.8 μm for the other pores. The channel height (space between sieve and glass) is 1.0 mm, the channel width 10 mm and the length 9.0 mm.

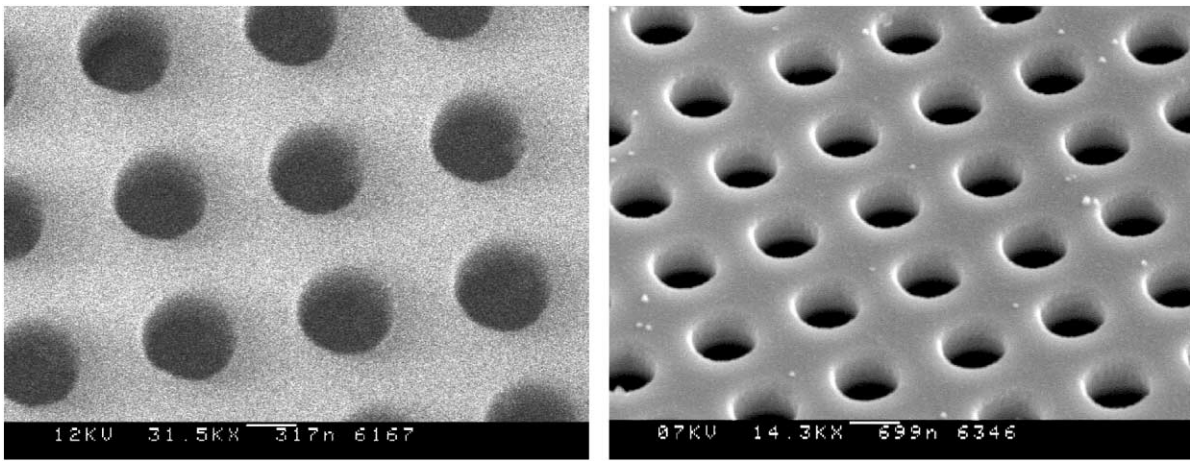

Fig. 3. Two of the microsieves with circular pores. Left: 0.55 μm pores, right: 0.80 μm pores.

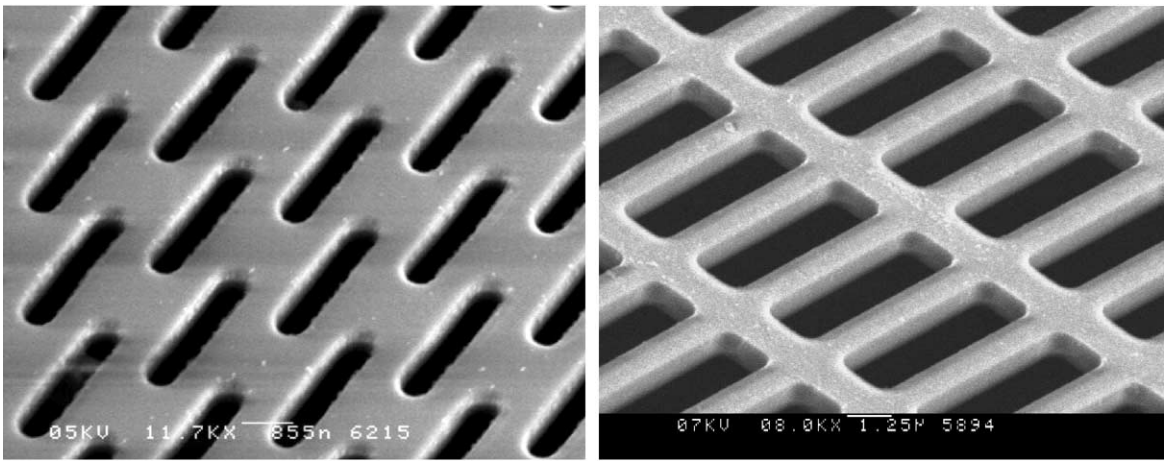

Fig. 4. SEM micrographs of the sieves with slit-shaped perforations. Left: 0.70 μm × 3.75 μm pores, right: 1.5 μm × 7.5 μm pores.

2.3. Lager beer

Lager beer was obtained direct from the brewery (Grolsche Bierbrouwerij Enschede). The beer was taken from two different stages of the brewing process: just before and just after centrifugation. During centrifuging the yeast content decreases by several orders of magnitude and also some aggregated proteins are removed.

2.4. Experiments

The experiments can roughly be devided into three subjects: membrane fouling, permeate turbidity and permeate flux.

Fouling was studied by observing the sieve surface through the microscope, while varying the filtration conditions. The most relevant results of these observations are described in Section 3. For conditions where cake-layer formation could largely be prevented by the crossflow and backpulses, permeate samples were collected and the turbidity was analysed at the brewery. The results of these turbidity analyses are given in Section 4. Finally, the sieves that produced the clearest permeates were used for flux measurements in long-run experiments. The results of these experiments are given in Section 5.

Unless otherwise specified all filtrations were performed at a temperature of 5°C, a crossflow of 50 l/h (with a resulting pressure drop along the feed channel of 0.030 bar) and an average transmembrane pressure of 0.15 bar. The backpulse pressure was −0.05 bar and the pulse duration 0.05 s. The pulse interval was varied, depending on the rate of pore obstruction, but was usually of the order of seconds.

3. Microscope observations: results and discussion

3.1. Yeast cells

For rough beer and a transmembrane pressure of 0.15 bar, all sieves were covered by a monolayer of yeast cells within a fraction of a second. This monolayer caused a flux decline by approximately one-order of magnitude. During a backpulse it was observed that all yeast cells were removed from the surface.

When lowering the transmembrane pressure, the rate of pore obstruction declined fast (faster than the decline in pressure). Whereas initially the yeast cells arrived randomly at the surface, they showed a remarkable obstruction mechanism at lower pressures. Once a yeast cells was trapped, other cells were trapped in the upstream direction of this cell. An avalanche effect occurred and a monolayer of yeast cells grew in upstream direction. Fig. 5 shows this effect in a series of frames captured from videotape recorded during filtration.

On the open areas it was observed that yeast cells were trapped on a pore, but dragged away by

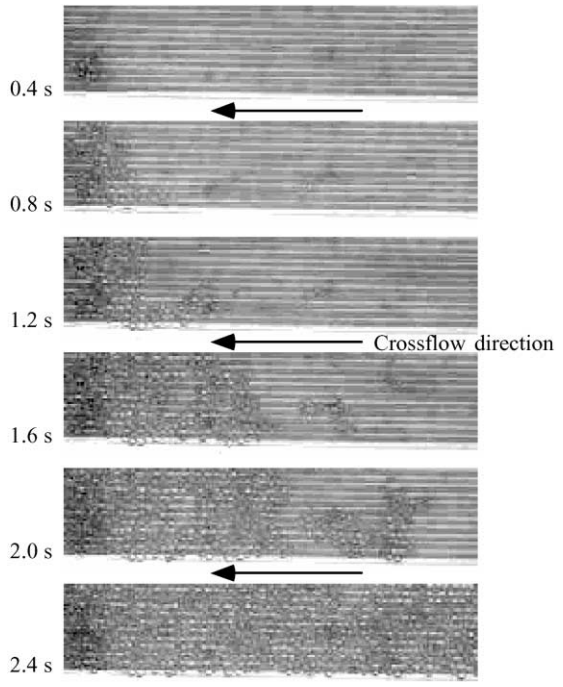

Fig. 5. Different stages of the pore-obstruction process. The numbers indicated denote the time that has passed since the last backpulse. The crossflow direction is from the right to the left.

the crossflow a fraction of a second after arrival. Apparently the transmembrane pressure was not large enough to keep the cells trapped. This phenomenon has been theoretically described by De Balmann et al. [10]. However, beer also contains other — smaller — particles like protein aggregates and cell fragments. These particles were often not dragged away and formed an obstruction for the yeast cells so that the avalanche effect could start. For even smaller transmembrane pressures the avalanche effect no longer occurred. An accurate description of the crossflow conditions necessary to keep the pores of a microsieve void of yeast cells was described in a previous paper [11].

3.2. Formation of flocks

When centrifuged beer was used it could be observed that many small particles (of the order of the pore size) were trapped on the pores. Like the yeast cells these particles could be removed with a backpulse. However, very few particles were not removed. They appeared to be attached to the membrane by transparant ‘wires’ with a length of approximately 1 μm. The wires were stuck in the pores or on the surface between the pores. The particles obstructed the pores, but during a backpulse they were lifted off the surface. After the pulse they immediately obstructed the same pores again. Such particles appeared to be able to catch other particles and after a certain time (which varied from minutes to hours) the stuck particles had gathered a flock-like structure around them that was largely lifted off the surface during each backpulse. Especially along the edges of the membrane fields the flocks were numerous. They were usually attached at only a few points of the membrane surface. If such flocks would be allowed to dry after filtration in order to make an SEM micrograph, probably a structure like shown in Fig. 1 would appear. Fig. 6 shows two frames captured from videotape recorded after several hours of filtration.

In the first frame, there is a positive transmembrane pressure, whereas in the second frame the situation during a backpulse can be observed. Clearly visible is the loose attachment of the flocks to the membrane, as they are largely lifted off the surface. Fane [12] reported a similar fouling phenomenon during filtration with a Whatman Anopore membrane. He used ‘direct observation through the membrane’ (DOTM) and observed that the flocks grow by accumulating other flocks. In crossflow, the flocks reached critical size and then detached due to increased axial drag. We noticed a similar behaviour using our ‘direct observation *on* the Membrane’ (DOOM) method, although the detachment is more an exception than a rule. Banplain et al. [13] did not use a direct observation method in their study of fouling mechanisms, but nevertheless, arrived at similar conclusions. Comparing permeate fluxes with classical filtration models, they concluded that the main phenomenon limiting the filtration of beer is the formation of aggregates of colloids that can form bridges over the pores by a mechanism of dendrite build-up. Such a dendrite build-up is confirmed by our observations of transparant wires connecting particles to the surface. Interesting is the fact that they filtered a clarified (kieselguhr-filtered) beer, but still found that on-pore fouling has a stronger influence on flow resistance than in-pore fouling.

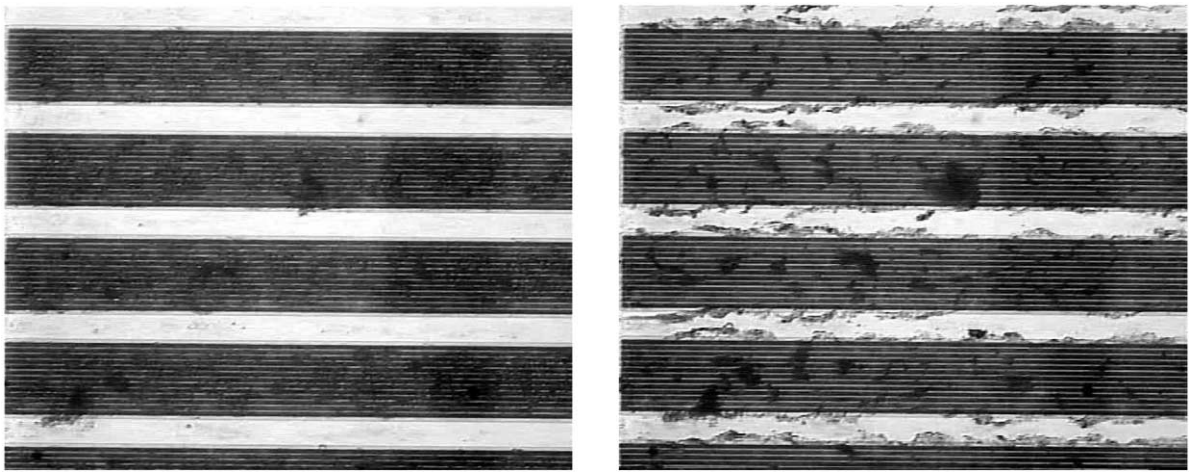

Fig. 6. Two frames captured from videotape showing loosely attached flocks. The frame on the left shows the situation during filtration and the frame on the right the situation during a backpulse.

The formation of flocks occurred for centrifuged beer as well as for rough beer. Fig. 7 shows a close-up photograph of a flock on a membrane with 1.5 μm × 7.5 μm slits during filtration of rough beer. The pictures were captured from videotape and represent the situation just before and just after a backpulse. The yeast cells are all removed, but the flock (it is hanging loosely over the unperforated area) remains.

The flocks seem to be composed of chill-haze proteins, which are large proteinaceous colloids formed at low temperature through the aggregation of hydrophilic proteins with a phenolic substance as the coagulating agent [6]. Protein aggregates may also be formed under influence of shear stress in the pump. Xu-Jiang et al. [14] show that the type of pump plays an important role in the aggregation of protein. They suggest that high shear stress in the pumps causes denaturation of proteins, which can subsequently form aggregates. This might be an explanation for our observation that during the run the concentration of

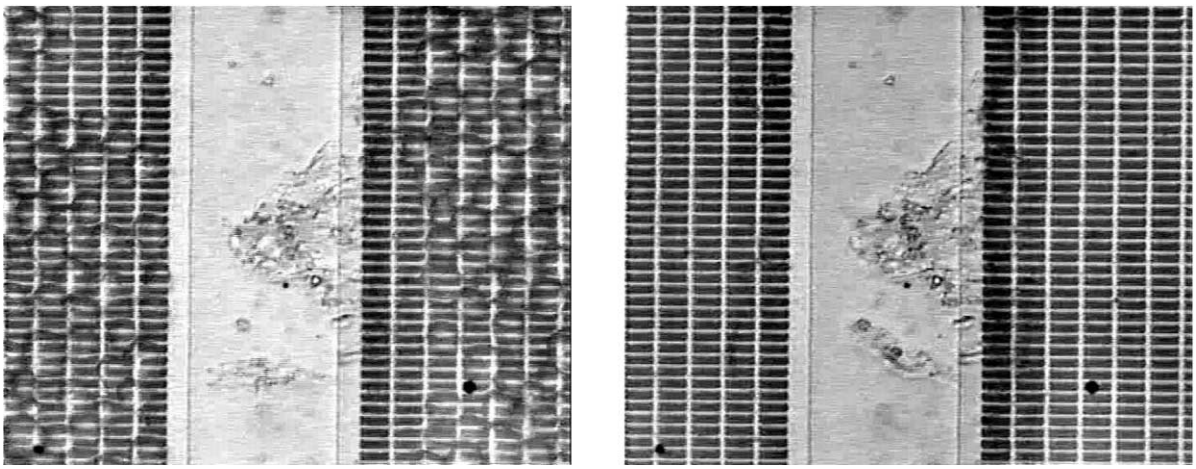

Fig. 7. Loosely-attached flock on a membrane with 1.5 μm × 7.5 μm slits during filtration of rough lager beer. The picture on the left shows the situation during filtration and the picture on the right immediately after a backpulse. Crossflow direction is from the right to the left.

particles increased stronger than might be expected from concentrating the feed.

In order to know whether other materials than proteins participate in the fouling process, we subjected the flocks to two standard tests that are available for breweries. The tests were used to detect the presence of ß-glucans and starch, but the results were negative for both components.

As detachment of the flocks sometimes occurred under influence of the crossflow-drag force, it should be possible to exploit this effect by applying a stronger crossflow. A successful method turned out to be a short (several seconds) 'crossflow boost' to 130 l/h (compared to 50 l/h under normal filtration conditions). In combination with gas sparging this method gave even better results. Nearly all flocks could be removed. The method only works if the permeate flow is temporarily stopped, so that the flocks are not pushed onto the membrane by the transmembrane pressure. Periodic stopping of the permeate flow in combination with air bubbling was earlier described by Tanaka et al. [15]. Using this method they found a significant increase in flux for a suspension of baker's yeast.

3.3. In-pore fouling

As mentioned before, the sieves consist of several rectangular membrane fields. We constructed sieves with half of these fields placed perpendicular and the other half parallel to the crossflow. Besides the formation of flocks on top of the pores, both kinds of fields showed an irreverible fouling that began on the downstream side of each field. The fouling layer slowly grew in the upstream direction. A consequence was that the perpendicular fields were largely blocked at the end of a run, whereas the parallel fields were largely open. The fouling layer was not well visible, but it could be observed that it was inside the pores. The blocked areas could be indirectly observed in case there was a gas bubble behind the membrane field. During a backpulse the clean areas lightened up as the bubble touched the membrane, whereas the fouled areas remained dark because the permeate could not be pushed through the pores and, hence, the bubble could not touch the membrane. Another indirect way to see the fouled areas was the capturing of particles. The fouled areas did not capture any particles. Fig. 8 shows two video frames recorded during a backpulse. The dark spots indicate the place of the fouling layer. During filtration these spots were hardly visible.

On some spots also the fields parallel to the crossflow suffered from the in-pore fouling. These were exactly the spots where flocks were observed earlier. Apparently, underneath these flocks a permanent fouling layer can grow and, therefore, attention should be paid to detach them frequently. The process of a

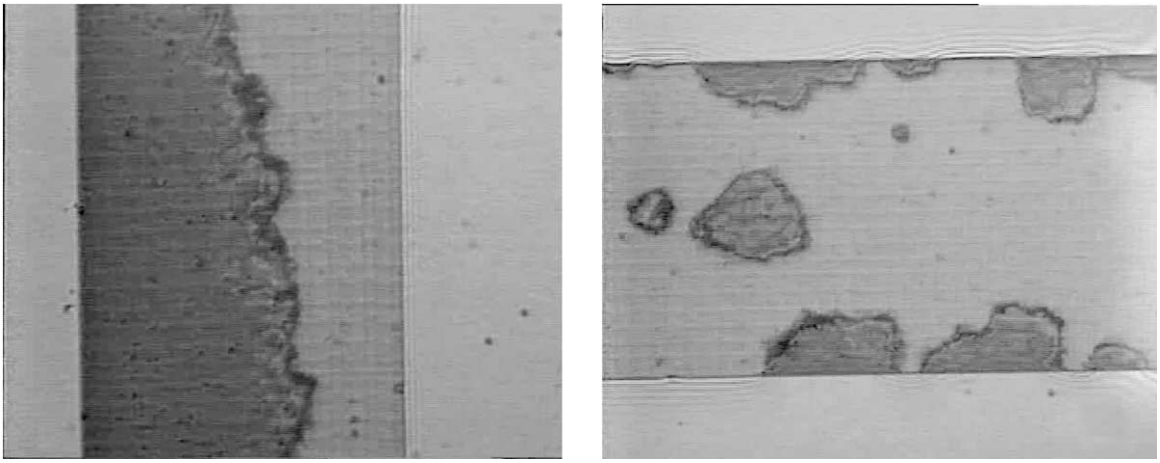

Fig. 8. Permanently blocked pores (the dark regions) made visible by a gas bubble underneath the membrane during a backpulse. The crossflow direction is from right to left.

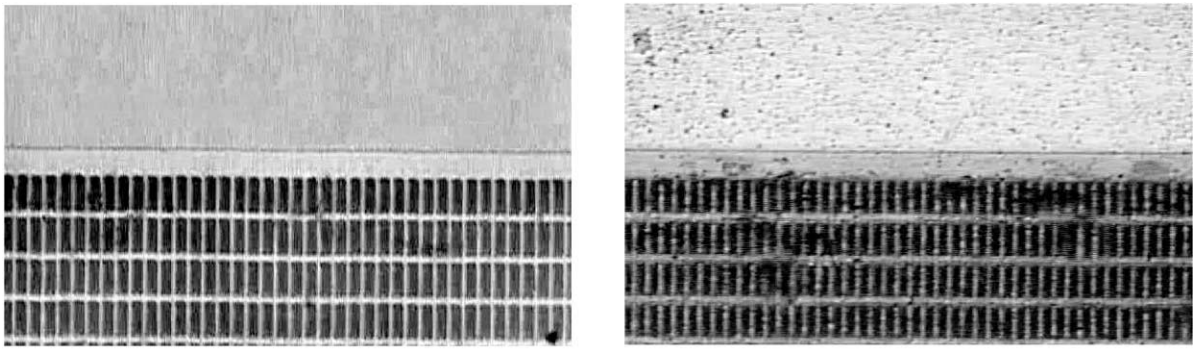

Fig. 9. Precipitation on the surface after cooling down from 5 to −1°C.

permanently fouled area that slowly grows in up-stream direction appeared for all sieves, regardless of pore size and shape.

3.4. Protein precipitation

During filtration the unperforated areas of the sieve remained clean. The beer components did not show a visible tendency to adhere to the surface. However, when the feed was cooled down from 5 to −1°C a severe precipitation of presumably proteins was observed. Small transparent particles (smaller than the pore size) precipitated in the pores and on the surface, herewith completely clogging the sieve. Fig. 9 shows a sieve before and after cooling down.

Heating up to the original temperature of 5°C made the layer dissappear again.

Precipitation could be prevented by leading the feed through a bypass along the sieve during cooling down, while closing the module. After the end temperature had been reached, the module was opened and precipitation on the sieve was not observed.

3.5. Cleaning

After filtration part of the fouling layer could be removed with warm water. Addition of standard enzymic membrane-cleaning agents removed even more of the layer, although the results varied quite strongly. With harsh chemical-cleaning methods it was possible to restore the original water flux, but the sieves had to be removed from the rig fur, such cleaning was done in order to protect the rig. We did not systematically investigate the cleaning process, but it is clear that further research on this point is needed.

4. Permeate haze: results and discussion

4.1. Flocculation in the permeate

Permeate samples collected during the first experiments showed a severe flocculation within a day, which made the haze results dependent on the time passed between collection and measurement. In the brewery, usually polyvinylpolypyrrolidone (PVPP) is added prior to kieselguhr filtration to remove the polyphenols. Polyphenols are known to form aggregates with protein. Addition of PVPP (0.15 g/l) prior to our experiments appeared to solve the problem: aggregation of protein in the permeate was no longer observed. In all next runs, PVPP was therefore added to the feed.

4.2. Haze values

For two batches of beer (rough beer and centrifuged beer) the permeate haze was determined. Samples of the feed collected before filtration were analysed as well. The results are listed in Table 1.

The haze values of the feed show that centrifuging removes a large part of the particles responsible for

Table 1
Haze of permeate and feed for rough beer and centrifuged beer

	Rough beer haze (EBC)	Centrifuged beer haze (EBC)
Feed	27.6	1.24
Slits (1.5 μm)	1.40	1.05
Circles (1.5 μm)	1.13	0.89
Circles (0.80 μm)	0.76	0.58
Slits (0.70 μm)	0.71	0.47
Circles (0.55 μm)	–	0.28

haze. After filtration the centrifuged beer gives significantly lower haze values than the rough beer.

The permeates produced with 0.70 μm slits and 0.55 μm circles are below the haze limit demanded by the brewery (0.50 EBC). The value of 0.28 EBC for the 0.55 μm pores is even comparable to what the brewer commonly obtains after kieselguhr filtration.

Similar investigations for ceramic membranes (Ceramem Corporation) on the effect of pore size on permeate turbidity were reported by Burrell and Reed [2]. They filtered two commercial rough beers, using pore sizes of 0.5, 1.0 and 1.3 μm. The 0.5 μm membrane resulted in 'exceptionally bright filtrates', typically 0.4 EBC and never above 0.65 EBC. The 1 μm membrane resulted in a haze between 0.55 and 0.75 EBC and the 1.3 μm membrane between 0.6 and 3 EBC.

5. Permeate flux

5.1. Experiments

Under the microscope it could be observed that yeast cells quickly obstructed the pores, thus, causing a fast flux decline. In order to prevent this, low transmembrane pressures, high crossflow velocities or high backpulse frequencies are necessary. As the permeate of the centrifuged beer was significantly clearer than that of the rough beer, and as the permeate flux of centrifuged beer will be significantly higher, we decided to use centrifuged beer for the flux measurements. The sieves with 1.5 μm pores were no longer used, as the produced permeates were not much clearer than the feed.

The experimental conditions were chosen as specified in Section 2.4, and the backpulse interval was set at 1.0 s. During the filtration runs several crossflow boosts were carried out in order to remove the formed flocks.

5.2. Results and discussion

Fig. 10 shows the flux results of three different sieves over a period of approximately 10 h. The graph shows that the differences in fluxes between the three sieves are quite large. The lowest flux was obtained with the 0.55 μm pores: 2.21×10^3 l/m^2 h over a period

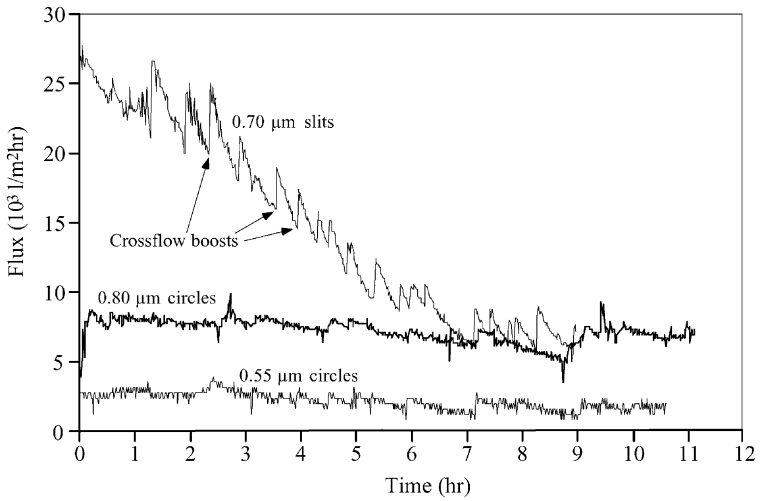

Fig. 10. Flux behaviour for filtration of centrifuged lager beer with three different microsieves.

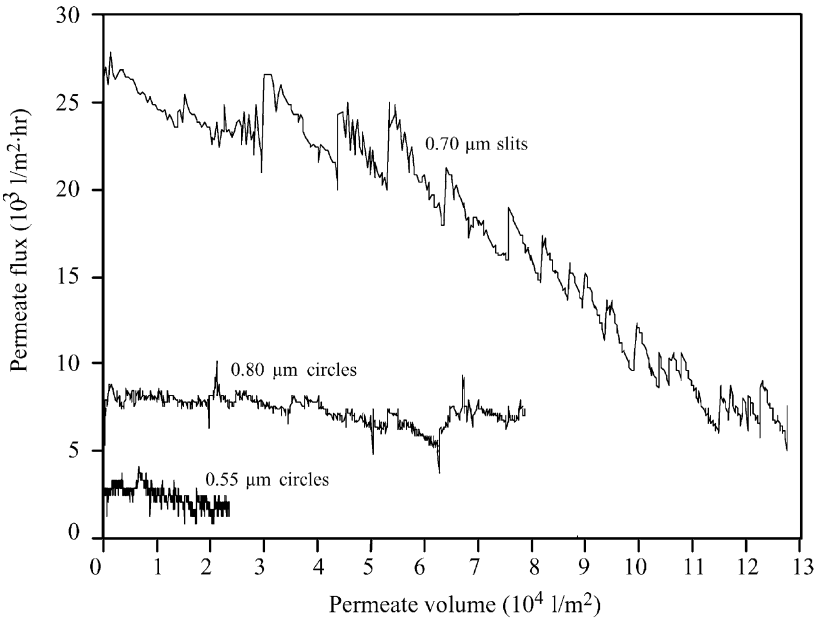

Fig. 11. Permeate flux plotted against accumulative permeate volume per square metre of sieve area.

of 10.5 h. This is still more than an order of magnitude larger than is commonly obtained for membrane filtration of lager beer. The sieve with slits produced the highest flux, but the rate of fouling was significantly larger than for the sieves with circular pores. For the sieve with slits it was difficult to remove the flocks with a crossflow boost: the peaks in the graph show that the flux after a crossflow boost did not reach the flux after the previous boost. The flocks appeared to be strongly attached to the membrane. For the 0.80 μm pores it was easier to detach the flocks and for 0.55 μm only half of the crossflow boost was sufficient to remove the flocks.

The feed-vessel volume of 2 l was not sufficient for the sieves with 0.70 and 0.80 μm pores. When the vessel was nearly empty, fresh beer was added during the experiments. A significant change in flux was not observed.

In order to be able to make a fair comparison between the different sieves regarding flux decline, the fluxes should be plotted as a function of permeate volume rather than time. Such a plot is shown in Fig. 11.

Now the flux decline of the sieve with slits looks less severe in comparison with the other sieves. The horizontal axis represents the volume of beer that passed through the membrane, which is a better measure for the fouling probability than time.

Besides permeate flux, the water flux of the sieves was measured before each filtration. The results are listed in Table 2, together with some other relevant results of the three long-run experiments.

Table 2
Results of the three long-run experiments

	Porosity (%)	Average beer flux (l/m^2 h)	Initial beer flux (l/m^2 bar h)	Water flux at 20°C (l/m^2 bar h)	Permeate haze (EBC)
Slits (0.70 μm)	31	14.3×10^3	18×10^4	18×10^5	0.46
Circles (0.80 μm)	22	7.24×10^3	5.6×10^4	5.8×10^5	0.58
Circles (0.55 μm)	24	2.21×10^3	1.8×10^4	4.7×10^5	0.23

For the 0.70 μm slits and 0.80 μm circles it appears that the initial beer flux is approximately a factor of 10 smaller than the water flux. This large difference has several causes. The viscosity of the beer at 5°C is typically 3 Pa s, which is three times larger than the viscosity of water at 20°C. Another cause for the large difference is due to the backpulses that occur every second (water fluxes were measured without back pulses). Finally, in-between two backpulses the membrane is partially blocked with particles like aggregated proteins. This is one of the reasons for the even larger difference (a factor of 26) between water and beer fluxes for the sieve with 0.55 μm pores. The smaller pores retain more particles and will, thus, cause a faster flux decline in-between two backpulses. It may, therefore, be effective to increase the backpulse frequency for the sieve with 0.55 μm pores.

5.3. Concentration factor

During the runs the permeate was not recycled. This implies that the beer was concentrated during filtration. Due to the large dead volume of the set-up the maximum concentration factor was approximately 4. In order to investigate the flux behaviour as a result of increasing concentration, the tubes and heat exchanger were replaced by smaller ones, thus, obtaining a smaller dead volume.

A concentration experiment was carried out with the 0.70 μm slits for almost identical conditions as before. The only adjustment was (besides the smaller dead volume) the backpulse period. It was decreased by a factor of 2–0.5 s in order to anticipate the expected increase in pore-blocking rate. The results are shown in Fig. 12.

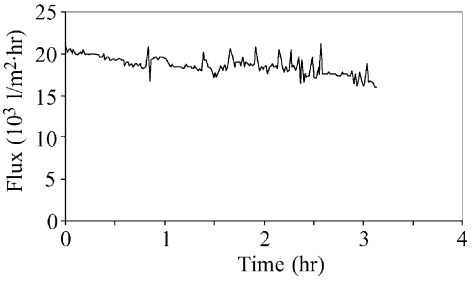

Fig. 12. Concentration of lager beer with 0.70 μm slits. The concentration at the end of the run was 12.

After concentration by a factor of 12, the decrease in flux was only 13%. The average flux over a period of 3.5 h was 18.3×10^3 l/m^2 h. Combined with the crossflow of 50 l/h this results in an average ratio of permeate flux over crossflow of 1.1%.

6. Scaling up

We have shown that on a 0.3 cm^2 sieve the fouling process may largely be controlled by periodic backpulses and crossflow boosts. During such boosts the pressure drop across the module rises from 0.03 bar to approximately 0.3 bar. For scaling up it is likely that 6 in. wafers will be used for the microsieve production. The channel length will then increase by a factor of 17 compared to the sieves that were used in this work. For such long channels a crossflow boost would create a pressure drop of approximately 5 bar, which will cause a high transmembrane pressure at the inlet of the channel, thus, hindering detachment of the flocks. The pressure drop across the channel can be decreased by increasing the channel height. However, this will result in a strong increase of the required crossflow energy. It may, therefore, be necessary to divide the crossflow channel into several short parallel channels with the help of a spacer that is placed above the sieve. In this way the channel height can remain low.

7. Conclusions

A crossflow-microfiltration rig was built in order to study fouling of microsieves through in-line microscope observations. The fouling process started with the formation of loosely attached flocks on the surface, gradually followed by in-pore fouling underneath these flocks. Strong attachment of the flocks to the sieve surface was prevented by applying a periodic backpulse. Most of the flocks could be removed by a strong temporary increase of the crossflow, if necessary in combination with gas sparging. Using this method, filtration intervals of approximately 10 h were achieved with average fluxes of more than two-orders of magnitude higher than is commonly obtained with membrane filtration. Using a sieve with a 0.55 μm pore size a permeate haze of 0.23 EBC was obtained during 10.5 h of filtration at an average flux of $2.21 \times$

10^3 l/m^2 h. A sieve with slits of 0.70 μm × 3.0 μm produced a less clear permeate (0.46 EBC), but the average flux over 9 h was huge: 14.3×10^3 l/m^2 h. In another run over 3 h, the feed was concentrated by a factor of 12, while the permeate flux decreased by only 13%.

A good temperature control appeared to be an important factor in keeping the sieves clean. Cooling down of the beer in the rig should be avoided, as this led to precipitation of presumably protein on the surface and inside the pores.

The experiments were performed on small (0.3 cm^2) microsieves. Scaling up will lead to larger pressure drops across the crossflow channel. This problem may be avoided by dividing the channel in several short channels with a spacer. The overall results are very promising, but both the issue of scaling up and chemical cleaning need further investigation.

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