Microfiltration Studies with a Modified Membrane Filterability Procedure


ABSTRACT

For the efficient operation of microfiltration plants, three areas need to be addressed. This paper, which addresses the first of these, proposes a modified method for measuring the membrane filterability of beer. The method is a modification of the method proposed earlier by Meier (1992) which essentially involves measuring the maximum quantity of beer that can be filtered through a particular membrane filter. The effect of various parameters (such as beer particle loading, gelling substances, isinglass finings), and processes (such as centrifugation, prefiltration, and dilution) are discussed. It is generally found that the reduction of particle loading and gelling substances and the use of isinglass finings improve the beer membrane filterability.

By recognizing that the “true” membrane filterability of the beer is the filterability in the absence of particle effect, pretreatment methods were developed for pre-bright beer stage samples. The practical implications from this are:
1. the membrane filterability of beers can be predicted upstream of the bright beer stage, allowing for corrective actions to be taken; and
2. raw materials (malt) and exogenous enzymes can be evaluated quickly, accurately and more importantly, using small samples.

INTRODUCTION

Three areas which need to be addressed for the efficient operation of a dead-end microfiltration plant are the membrane filterability of the beer, the cleaning/regeneration of the filters and the optimum choice of the filter train and membrane material. This paper addresses the first of these. If the beer is highly filterable then the length of filter run before needing regeneration/cleaning is extended. The cleaning/regeneration of the filters is just as important since for every successful cleaning the filters can be reused for another cycle. Finally, the choice of filter train and membrane material will affect both the membrane filterability of the beer and the cleaning regimes the filters can take.

All membrane filterability tests are based on performing filtration under a given constant upstream pressure, using a membrane filter of a particular diameter with a defined absolute rating. The original test (Esser, 1972) involved a five-minute filtration using cellulose acetate membrane of 16 cm² surface area with 0.2 µm absolute rating, and a 200 kPa upstream pressure. Eyben and Duthoy (1979) and Siebert et al. (1984) used the test to predict wort lautering and beer diatomaceous earth (DE) filtration performances. Later, Reid et al. (1990) and Meier (1992, 1994) used the membrane filterability test for microfiltration optimization and scale up.

The term “beer filterability” has been used almost exclusively to refer to the DE filterability of beer. With an increased application of membrane filtration in beer production, it would be advantageous to have a test which differentiates between the DE filterability and the membrane filterability of beer. A method for evaluating the DE filterability of beer is available (Lim et al. [1992], Timmerman [1994]), and there is anecdotal evidence which suggests that beers with DE filterability (using the method of Lim et al. [1992]) less than or equal to 400 sL⁻² would generally present little or no problem during DE filtration (using Standard Supercel) in the plant.
Quantifying Membrane Filterability

The result from a membrane filterability test is generally reported either as \( V_{\text{max}} \) value or as a plugging index. \( V_{\text{max}} \) is defined as the maximum volume of liquid which can be filtered through the membrane filter. \( V_{\text{max}} \) value can be extracted from time vs cumulative volume data from a membrane filterability test (\( V_{\text{max}} \) test). The cumulative volume or weight data when plotted against time will fit the following equation if the filters blocks by the “gradual pore plugging” (Meier, 1992) or the “absorptive sequestration” (Hermans and Bredee, 1936) model:

\[
V = \frac{t}{(At+B)}
\]

where \( t = \text{time (minutes)} \)

\( V = \text{Volume collected up until } t \text{ (Litres)} \)

\( A = \text{a constant related to } V_{\text{max}} \)

\( B = \text{a constant related to initial filtration rate} \)

Dividing both top and bottom by \( t \),

\[
V = \frac{1}{(A + \frac{B}{t})}
\]

As time approaches infinity, \( V \) approaches \( V_{\text{max}} \), the maximum volume of liquid which can be filtered.

\[
V_{\text{max}} = \frac{1}{A}
\]

Using equation (1), at time = 0 minute, initial flowrate, \( Q_{\text{init}} \), can be calculated from:

\[
Q_{\text{init}} = \frac{1}{B}
\]

Rearranging equation (1),

\[
\frac{t}{V} = At + B
\]

By regressing \( t/V \) vs \( t \), the slope and y-intercept of the line are related to \( V_{\text{max}} \) and \( Q_{\text{init}} \) by equations (3) and (4). Esser (1972) used a simple two point method to extract the \( V_{\text{max}} \) value:

\[
V_{\text{max}} = \frac{3V_2}{(5V_2 - V_5)}
\]

where \( V_2 = \text{Volume collected at 2 minutes, L} \)

\( V_5 = \text{Volume collected at 5 minutes, L} \)

Cuno (1972) suggested the use of the above equation for \( V_{\text{max}} \) value extraction, but with the filtration carried out at 1 bar using 25 mm diameter Nylon 66 filter of 0.45 or 0.65 \( \mu \text{m} \) pore rating. With the advent of faster and cheaper computers, more recent workers (Reid et al., 1990; Meier, 1992, 1994) regressed more data points to obtain a statistically more significant \( V_{\text{max}} \) value. More complex model of liquid flow through membrane filters have recently been proposed by Jackson (1995).

The other approach is to express the membrane filterability in terms of a Plugging/Filterability Index (\( PI/Fl \)). There are several definitions of \( PI/Fl \). In the wine industry, the so called “wine filterability index” which is also known as “indice de colmatage-IC” is often used:

\[
Fl = \frac{T_{400}}{2T_{200}}
\]

where \( T_{400} = \text{time to filter 400 mL of sample, 0.01 minute} \)

\( T_{200} = \text{time to filter 200 mL of sample, 0.01 minute} \)

This index is obtained by performing the filtration using a 25 mm diameter, 0.65 \( \mu \text{m} \) filter membrane (Nylon 66) under 2 bar pressure at ambient temperature. A value of 30 or below is usually accepted as an indication that the wine is suitably filterable (1). Reid et al. (1990) adopted this index for beer but used 0.45 \( \mu \text{m} \) PVDF membrane and expressed the index in seconds. Reid claimed that beer \( Fl \) of around 10 was needed for economical beer filtration. Assuming that the filtration data can be described by equation (1), then \( Fl \) can also be calculated from \( V_{\text{max}} \) and \( Q_{\text{init}} \) data by the following equation:

\[
Fl = \frac{4.8V_{\text{max}}}{Q_{\text{init}}(V_{\text{max}}-0.4)(V_{\text{max}}-0.2)}
\]

Another filterability index sometimes used is the silting index \( (SI) \) or silt density index \( (SDI) \). It is defined as:

\[
SI = 100 \left( \frac{1 - \frac{\Delta T_0}{\Delta T_f}}{T_f} \right)
\]

where \( \Delta T_0 = \text{the time required to collect the first 100 mL of filtrate, seconds} \)

\( \Delta T_f = \text{the time required to collect the last 100 mL of filtrate, seconds} \)

\( T_f = \text{the starting time at which the last 100 mL of filtrate was collected, minute} \)

The last 100 mL collected is when \( \Delta T_0 / \Delta T_f \leq 0.2 \). Pall used this index mainly for water filtration, the test is normally performed using 47 mm diameter cellulose acetate filter with 200 kPa upstream pressure.
Redefinition of Membrane Filterability or \( V_{\text{max}} \)

It is clear from the foregoing discussion that due care must be taken in comparing the membrane filterability values, be it in the form of \( V_{\text{max}} \) or plugging index data. There is a need therefore to find an agreed standard method for membrane filterability determination. Plugging indices are useful for cases where the filtration data do not fit the “gradual pore plugging” model. However, for cases where the model fits the data, \( V_{\text{max}} \) and \( Q_{\text{init}} \) can provide useful data for scale up purposes if the same filter material is used for the plant scale filtration (Meier, 1992).

The membrane filter in the microfiltration plant will generally encounter essentially “particle free” beer. It would be logical, therefore, to redefine \( V_{\text{max}} \) as the maximum volume or weight of essentially particle free beer which can be filtered through a given membrane filter under 200 kPa upstream pressure. “Essentially particle free” is defined as low enough particle loading such that small changes in the particle loading will not affect the membrane filterability of the beer.

To perform \( V_{\text{max}} \) determination using the Meier’s method would require a large volume of sample, of the order of several litres. The modified method only requires 0.5 L per determination or 1 L for duplicate runs.

![Figure 1. Schematic Diagram of the Membrane Filterability Test Equipment.](image)

**Experimental**

**Membrane Filterability Equipment**

The proposed membrane filterability method employs commercially available Pall Wine Filterability Apparatus or Cuno Membrane Plugging or Silting Index Apparatus. A schematic diagram is shown in Figure 1. The filterability apparatus consists of a liquid holding chamber with capacity of about 450 mL which is connected to a 25 mm filter holder by a ball valve. The membrane filters used are made from Nylon 66 with absolute pore rating of 0.65 \( \mu \)m. This filter was chosen because it was conveniently available at the time and fortuitously provided good repeatability. The use of another membrane, 0.45 \( \mu \)m absolute rated PVDF filter was later investigated. These filters gave generally higher \( V_{\text{max}} \) values, but showed much poorer repeatability. It was noted that for a particular batch of the PVDF filters the standard deviation of their dry weight (2.2%) was larger than a particular batch of Nylon 66 (0.35%) filters. This may have an impact on the repeatability of the method, but the matter was not investigated further.

The \( V_{\text{max}} \) tests are performed by filling the liquid holding chamber with cold (0-5°C), degassed beer, pressurizing the chamber to 200±5 kPa, prewetting the filter by quickly opening and closing the ball valve, and starting filtration after taring the balance. The balance readings are recorded every 30 seconds until either the chamber is empty or the change in weight during the latest time period is less than 35% of the first balance reading. This last condition was introduced to ensure that most of the relevant data has been included in the regression. It can also reduce the length of the experiment, especially for beers with very low \( V_{\text{max}} \) values.

The \( V_{\text{max}} \) values were extracted by regressing the \( t/N \) vs \( t \) data as described above, but ignoring the first three data points. The first three data points from such plots usually deviate significantly from the regression line as shown in Figure 2. The correlation coefficient from the regression must be greater than or equal to 0.99 for the data to be considered valid for \( V_{\text{max}} \) value determination. Experiments were performed at least in duplicate and the average \( V_{\text{max}} \) values obtained are reported.

The maximum \( V_{\text{max}} \) value accurately obtainable using this method is around 1.3 L.

**Particle Counting**

A Coulter Counter and Multisizer coupled with Accucomp Software (Coulter Electronics, USA) was used to count the number of particles. Determinations were made using a 70 \( \mu \)m diameter aperture tube. The same procedure as described earlier (Lim et al., 1992) was used.

![Figure 2. Typical Regression of \( t/N \) vs \( t \) data for \( V_{\text{max}} \) Value Extraction.](image)

\[ V_{\text{max}} = 0.78 \text{ Litres}, \ Q_{\text{init}} = 0.097 \text{ L/min}, \]

Correlation Coef. = 0.998.
Sample Pretreatment

For beer samples post diatomaceous earth filtration stage, the pretreatment used was to degas the sample by carefully pouring the beer into a beaker. The beaker was then covered and the beer left in the refrigerator until no gas evolution could be detected. This normally took between 20 minutes to 1 hour. Foaming of the sample should be minimized to avoid the possible formation of dried foam flakes which will interfere with the \( V_{\text{max}} \) determination.

Beer samples pre-diatomaceous earth filtration stage were pretreated to reduce the particle loading. Two pretreatment methods were investigated, prefiltration using Cuno Zetaplus 10S grade pad filters and centrifugation. The centrifuge used was Jouan model CR411 which has refrigeration and swing out rotors. \( V_{\text{max}} \) values are classified by the processing stage from which the samples were taken. In this work, three different processing stages are considered: End of Fermentation (EOF), End of Storage (EOS) and Bright Beer (BB).

RESULTS AND DISCUSSION

In order to gain confidence with the modified \( V_{\text{max}} \) method, several preliminary investigations were carried out using bright beer samples. This includes the repeatability of the method and the effect of temperature and pressure.

Bright Beer (BB) \( V_{\text{max}} \)

Repeatability

The repeatability/precision of the method was determined by performing replicate filtrations with various bright beer samples. The standard deviations from such work ranged from 0 to 11% of the mean values. The average standard deviation being 6% of the mean values. This result is in good agreement with Siebert (1984) who obtained a standard deviation of 5.5% of the mean value for low haze beer using cellulose acetate membranes.

Effect of Temperature on Bright Beer \( V_{\text{max}} \)

The results confirm the fact that the diatomaceous earth filtration has been optimized. The total particle count of the various bright beers investigated ranged from 5,000 to 50,000 per mL. These were essentially permanent haze particles. The effect of centrifugation at 2500 x g for 20 minutes is shown in Table 1. It is clear that centrifugation had only a minor effect on the membrane filterability of the bright beer samples. The results confirm the fact that the diatomaceous earth filtration produced “essentially particle free” beer.

Effect of Pressure on Bright Beer \( V_{\text{max}} \) Values.

The effect of upstream pressure on \( V_{\text{max}} \) value was investigated using two different bright beer samples and the results shown in Figure 4. The \( V_{\text{max}} \) value increased as the pressure was increased from 140 kPa to 300 kPa. Since plant scale microfiltrations are normally carried out to a maximum of 200 kPa, the membrane filterability test were performed using 200 kPa upstream pressure. Between 195 and 205 kPa, the \( V_{\text{max}} \) values were well within the precision range above.

Particle Loading

Bright beer by definition should be “essentially particle free” if the diatomaceous earth filtration has been optimized. The total particle count of the various bright beers investigated ranged from 5,000 to 50,000 per mL. These were essentially permanent haze particles. The effect of centrifugation at 2500 x g for 20 minutes is shown in Table 1. It is clear that centrifugation had only a minor effect on the membrane filterability of the bright beer samples. The results confirm the fact that the diatomaceous earth filtration produced “essentially particle free” beer.
Table 1
Effect of Centrifugation on Bright Beer Vmax Values

<table>
<thead>
<tr>
<th>Sample</th>
<th>Vmax (L) As is</th>
<th>Vmax (L) Centrifuged</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beer 2a</td>
<td>0.73</td>
<td>0.79</td>
</tr>
<tr>
<td>Beer 2b</td>
<td>0.63</td>
<td>0.57</td>
</tr>
<tr>
<td>Beer 2c</td>
<td>0.62</td>
<td>0.65</td>
</tr>
</tbody>
</table>

Table 2
Comparison of End of Storage Beer Pretreatment Methods to Reduce Particle Number

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>As is</th>
<th>Prefiltered through Cuno 10S</th>
<th>Centrifuged at 2500 x g for 10 minutes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beer 1A</td>
<td>0.08±0.01</td>
<td>0.34±0.10</td>
<td>0.37±0.03</td>
</tr>
<tr>
<td>Beer 1B</td>
<td>0.06±0.01</td>
<td>0.31±0.11</td>
<td>0.32±0.02</td>
</tr>
<tr>
<td>Beer 1C</td>
<td>0.10±0.01</td>
<td>0.27±0.03</td>
<td>0.64±0.01</td>
</tr>
<tr>
<td>Beer 1D</td>
<td>0.06±0.01</td>
<td>0.24±0.05</td>
<td>0.29±0.01</td>
</tr>
<tr>
<td>Beer 1E</td>
<td>0.22±0.05</td>
<td></td>
<td>0.53±0.05</td>
</tr>
</tbody>
</table>

Figure 5.
Bright Beer Vmax vs Total β-Glucan (Calcoflour Method).
No Correlation Observed in the Range Investigated.

Gelling Substances

It was found that total β-glucan (Calcofluor method) content did not correlate with the bright beer membrane filterability as shown in Figure 5. Kruger et al. (1989) suggested that it is the gelling fraction of the beta-glucan which contributes to the problems in filtrations.

There are two methods for the isolation of gelling substances in beer. These are the freeze-thaw (Takayanagi et al. 1969) and the kieselguhr absorption (Eiselt and Kruger 1993) methods. Excessive shearing of the beer using a laboratory homogeniser was used by Letters (1977) to accelerate the formation of beta-glucan gels. In this work, gelling substances were isolated by the freezing and thawing method. The term gelling substances is used because it was found that the gelling material also contained protein and other polymers of five ring sugars.

Figure 6 shows the effect of gelling substances (expressed as β-glucan gel) concentration on the bright beer membrane filterability. These results agreed with the findings of Kruger et al. (1989). The most significant point here is that very little quantities of the gelling substance were required to reduce the membrane filterability by about 50%.

End Of Storage (EOS) Vmax

Vmax tests are generally performed on bright beer samples to determine whether the beer has good membrane filterability (high BB Vmax value). At this stage of beer production, it would be too late to make any corrective action if the beer is found to have low BB Vmax value. It would, therefore, be useful if the Vmax test can be performed at an earlier stage of beer production. An obvious choice would be to perform the Vmax test at the end of storage (lagering) or at the end of fermentation stage.

Reducing particle loading

Siebert (1984) investigated the effect of time on the membrane filterability of storage beer during lagering. He found that it increased moderately. The membrane filterability measured by Siebert would have been affected by both particulate and gelling substances. Depending on the treatment given to the beer during transfer from fermentation to storage, different particulate levels
would be reached at end of storage (Lim et al., 1992). In line with the definition of membrane filterability as essentially for “particle free” beer, methods for achieving this for storage beer were investigated.

Prefiltration vs Centrifugation of Storage Beer for EOS Vmax Determination

Two pretreatment methods for reducing particle loading in EOS beers were investigated. The first is the prefiltration of the samples through Cuno Zetaplus 10S grade pad filter, the second centrifugation at 2500 x g for 10 minutes.

The results are shown in Table 2. Without sample pretreatment to remove particulate (As is data), there is very little difference between the five samples. The pretreatments increased the EOS Vmax by at least 3-fold. Between the two pretreatment methods, the prefiltration method produced much more variable EOS Vmax data than the centrifuged samples. Although seemingly simpler, the prefiltration method suffers from the broad nominal pore rating specification of the depth filter (0.8 to 4 μm). Depending on the initial particle loading, the prefiltration can be between a few minutes to over an hour. Another problem with the prefiltration was the release of filter aid from the filter pads. In contrast, the centrifugation method is more consistent and in the long run will cost less.

In order to ascertain that the centrifugation speed was the appropriate speed, the effect of centrifugation speed on the EOS Vmax was examined. Figure 7 confirmed that centrifugation at 2500 x g for 10 minutes was sufficient in minimizing the effect of particulate loading from the storage beer. As the centrifugation speed is increased the EOS Vmax approached an asymptotic value (within the accuracy of the Vmax method). Centrifugation at lower speed but longer time is another alternative which can be used but was not investigated. It suffices to say that the total particle count decreased from about 2x10^5 to about 3x10^4 per mL, which is in the same range as bright beers from the same brewery.

Correlation between Bright Beer and EOS Vmax

The main aim of investigating the determination of EOS Vmax was to enable prediction of bright beer Vmax at an earlier stage of production so that corrective action can be taken. The natural progression from the work just described would be to correlate the BB Vmax against the EOS Vmax. Using EOS and bright beer samples from a particular production plant, the two sets of data were correlated and the results shown in Figure 8. The EOS Vmax values were determined by centrifuging the beer at 2500 x g for 10 minutes, while the BB Vmax values were determined on the samples as is. For practicality and simplicity, the regression line was forced through the origin, resulting in the following equation:

$$BB_{Vmax} = 1.6 \times EOS_{Vmax}$$

with a correlation coefficient of 0.97.

The factor 1.6 will probably apply only to the particular brewery where the beer samples were taken from. This is
because there are several different processes occurring between the end of storage stage to the bright beer stage. These may include dilution (if the brewery uses high gravity brewing), carbonation, trimming, dosing of various stabilizers and filtration. The most relevant processes in terms of particulate and gelling substances are dilution and filtration, and the order in which these are performed. In the brewery where the samples were obtained, the EOS beers were diluted by 25% of their original volume before filtration. It is interesting to note that the 25% dilution prior to diatomaceous earth filtration resulted in 60% increase in the membrane filterability of the beer. This additional increase in membrane filterability could either be caused by the dissolution of chill haze particles and gelling materials or the absorption of the gelling materials on to the diatomaceous earth.

**Effect of dilution on EOS Vmax**

The effect of diluting EOS beer before and after diatomaceous earth filtration were investigated. In the first set of experiments, EOS beer was filtered through diatomaceous earth and subsequently diluted with varying amounts of deoxygenated water. The membrane filterability of the beers was determined and compared. In the second set of experiments, the EOS beer was diluted, centrifuged as described above and the membrane filterability determined.

Figure 9 compares the results on the effect of dilution after diatomaceous earth filter outlet. These samples were centrifuged at 2500 x g for 20 minutes and membrane filterabilities determined. The results are shown in Table 3. The effect of diluting the EOS beer was just as observed with the laboratory dilution of the EOS beer. DE filtration was observed to improve the membrane filterability of the normal gravity beer by 60% larger than expected. This could be due to the fact that the filter had just been put on recirculation and it was nearing the end of the filter run.

**Effect of enzyme addition on EOS Vmax**

The effect of adding commercially available β-glucanase enzyme to storage beer produced from worts which had not been dosed with β-glucanase was investigated. Table 4 shows the results. For the control beer (0 ppm enzyme addition), the Vmax of the “as is” sample did not improve even after centrifugation of the sample. In contrast, the Vmax of the beer treated with 20 ppm of enzyme increased dramatically upon centrifugation of the sample. These results clearly demonstrate that performing the membrane filterability test on particle laden beer will provide the wrong kind of information on the “true” membrane filterability of the beer. The non-enzyme treated EOS beer had very low membrane filterability. The enzyme complex increased the membrane filterability of the storage beer by up to 8 fold after 12 days storage at 0-2°C.

Aside from β-glucans, pentosans have also been blamed for beer and wort filtration problems. The effects of heating and xylanase enzyme addition on EOS Vmax were investigated using the same beer from the non-enzyme treated wort above. The EOS beer was divided into three portions, the first one was kept at 2°C (control), the other two portions were heated to 55°C (optimum temperature of the enzyme), one of which was dosed with an experimental “pure” xylanase enzyme preparation. The beers were heated for 3 hours, then cooled to 2°C and kept at this temperature for a further 5 days. EOS Vmax (pre-centrifuged samples) values and β-glucan concentrations were determined for the samples. The results are shown in Table 5.

The results show that heating alone increased the membrane filterability of the storage beer. A possible explanation can be found in the work of Letters (1995) who found that the beer gel dissolves when heated, disrupting the structure of the glucan gel. Once dissolved, the gel does not reform easily upon cooling unless subjected to freezing and thawing.

As expected, the xylanase enzyme further increased the EOS Vmax of the beer. The very small drop in β-glucan concentration of the beer is indicative that the xylanase enzyme is reasonably devoid of glucanase activity. McKechnie and O’Sullivan (1995)
also found that xylanase improved the filterability of difficult to filter beer through an Amicon stirred filtration cell.

**End of Fermentation (EOF) Vmax**

It is now possible to determine the membrane filterability of the beer at the end of storage stage. If this is low, then β-glucanase enzyme can be added to improve the beer. However, this would still cause delays in production. It is, therefore, desirable to find out the membrane filterability at the end of fermentation stage so that any deficiencies can be rectified without causing production delays. A suitable pretreatment method for end of fermentation beer is therefore needed. The end of fermentation stage is defined as the stage after the fermentation is completed and the beer chilled to stop the fermentation while at the same time allowing the yeast to settle.

**Effect of centrifugation speed on EOF Vmax**

The effect of centrifuging the beer at various speeds for 10 minutes on the EOF Vmax values was investigated. The results for three different beers are shown in Figure 10. The higher the centrifugation speed, the higher the Vmax value obtained. Because of the high solids concentration of the EOF beers, the EOF Vmax values did not reach any asymptotic values.

The laboratory centrifuge used was only capable of generating up to 3100 x g. To reduce wear on the centrifuge, it was decided to limit the maximum centrifugal force to 2500 x g. In order to minimize the effect of particulate on the EOF Vmax values obtained, longer centrifugation times were investigated. The

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**Table 5**

Effect of Heating and Xylanase on EOS Vmax of Beer From Undosed Wort

<table>
<thead>
<tr>
<th>Parameters</th>
<th>2°C Control</th>
<th>55°C Control</th>
<th>20 ppm Xylanase</th>
</tr>
</thead>
<tbody>
<tr>
<td>EOS Vmax (L)</td>
<td>0.08±0.01</td>
<td>0.28±0.01</td>
<td>0.45±0.08</td>
</tr>
<tr>
<td>β-Glucan (ppm)</td>
<td>147±5</td>
<td>145±5</td>
<td>136±5</td>
</tr>
</tbody>
</table>

**Table 6**

Effect of Isinglass Finings Addition and Contact Times on EOF Vmax

<table>
<thead>
<tr>
<th>Contact Time (Minutes)</th>
<th>EOF Vmax (20 minutes at 2500 x g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 ppm</td>
<td>0.27</td>
</tr>
<tr>
<td>10 ppm</td>
<td>0.42</td>
</tr>
<tr>
<td>20 ppm</td>
<td>0.40</td>
</tr>
<tr>
<td>5</td>
<td>0.42</td>
</tr>
<tr>
<td>30</td>
<td>0.41</td>
</tr>
<tr>
<td>50</td>
<td>0.52</td>
</tr>
<tr>
<td>150</td>
<td>0.50</td>
</tr>
<tr>
<td>960</td>
<td>0.26</td>
</tr>
</tbody>
</table>

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**Figure 10.** Effect of Centrifugation Speed on End of Fermentation Vmax.

**Figure 11.** Effect of Centrifugation Time (at 2500 x g) on End of Fermentation Vmax and Total Particle Count.

**Figure 12.** Correlation Between Bright Beer and End of Fermentation Vmax, End of Fermentation Beer Centrifuged at 2500 x g for 10 minutes.
Effect of isinglass finings addition on EOF $V_{\text{max}}$

Iisinglass finings are often used to accelerate the sedimentation process during storage. The effect of adding this material on the membrane filterability of EOF beers was investigated. The procedure was used to add the isinglass finings to the EOF beer in a measuring cylinder, cover the end of the cylinder with stretchable parafilm, and inverting the cylinder three times to mix the beer. After the indicated time periods, the beer was poured into 50 mL centrifuge tubes and spun at 2500 x g for 20 minutes. The supernatant was carefully decanted into the membrane filterability apparatus and the filterability determined.

The results are shown in Table 6. The membrane filterability improved with longer finings contact time, although between 5 and 30 minutes, and between 50 and 150 minutes, there were insignificant changes. The addition of isinglass finings improved the membrane filterability of the beer. Dosage rate (10-20 ppm) of the finings did not have any effect. In other experiments, the degree of improvement with finings was lower, but, in all cases, some improvement of filterability with finings was noted.

Correlation Between EOF and BB $V_{\text{max}}$

End of fermentation beer samples were collected together with the end of storage and bright beer samples by tracing through the history of the beer in the plant. The results for the correlation between EOS and bright beer membrane filterabilities are already reported above. During the survey, the EOF $V_{\text{max}}$ values were determined after isinglass finings addition (15 ppm) and centrifugation at 2500 x g for 10 minutes. The results are shown in Figure 12. Reasonably good correlation was found. Since the EOF $V_{\text{max}}$ were determined by centrifugation for only 10 minutes, the EOF $V_{\text{max}}$ values obtained were low.

CONCLUSION

The membrane filterability of beers as measured using the modified method is affected by the particle loading and the amount of gelling substances in the beer. The membrane filterability of the beer was redefined as the filterability in the absence of particle effect. The membrane filterability of beers upstream from the bright beer stage can be determined by pre-centrifuging the beers. Suitable pre-centrifugation treatment for end of storage beer is 10 minute (or longer) at 2500 x g. For end of fermentation beers, suitable centrifugation pre-treatment is 20 minutes (or longer) at 2500 x g. It was also shown that heat treatment and the addition of xylanase and $\beta$-glucanase into storage improved the membrane filterability of the beers. The addition of isinglass finings, the dilution of beer with water and the filtration of beer through diatomaceous earth increased the membrane filterability of the beer. Finally, the membrane filterability of the beer can be predicted at an earlier stage in the process using very little sample volume. This enables corrective action to be evaluated and implemented quickly. Other practical applications of the method are the evaluation of mashing enzymes and malts for microfiltered products.

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QUESTIONS AND ANSWERS

Q. What was your addition rate of Beta Glucanase to storage beer?
A. The rate of β-Glucanase addition to storage beer was 20 ppm.