Dealcoholization of Beer using Osmotic Membrane Distillation.

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ABSTRACT

In this study, the possibility of using osmotic membrane distillation in reducing the alcohol content of beer was investigated. Effects of various parameters such as temperature (30°C, 40°C), stirring speed (0,100rpm), and membrane pore size (0.2µm, 0.45µm) on the flux were studied. All the experiments were carried out in a membrane cell mode using hydrophobic polytetrafluoroethylene membrane. The design of experiment was done and the results obtained were analyzed using Design–Expert 8.0.2® software. It was observed that of all the factors investigated, increase in stirring speed had the highest effect on the flux with 21.4% increase followed by increase in temperature with 8.34% increase on flux. The least significant parameter was increase in membrane pore size with 8.6% decrease on flux. The statistical analysis done showed that the three factors had significant effect on the response variable which is the flux. The model adequacy check carried out led to the conclusion that the true response surface was explained by the linear model obtained.

(Keywords: osmotic membrane distillation, hydrophobic membrane, beer, flux, alcohol content)

INTRODUCTION

In recent years, considerable emphasis has been laid on the development and operation of processes for the production of beers with reduced alcohol content due to health and socio-economic implications. Commercial interest has also been stimulated by the potential for savings in tariffs on the reduced alcohol content of wines. Furthermore, wines with reduced alcohol content offer a number of potential social and health benefits for consumers. Social benefits may include improved productivity and function after activities involving alcohol consumption, lower risk of prosecution or accident while driving and the health advantages may include reduced calorie intake and decreased risk of alcohol related diseases.

Some people may guess that non-alcoholic or low-alcohol beers would be made by using much less malt, on the contrary, reduction in malt results in truly bland taste. Remember that the malt not only contributes to the fermentable sugar content of beer but also to the overall taste of the beer. Some people actually consume beer due to the intoxicating effect of the alcohol while others consume it due to the main flavor/fragrance of the beer resulting from the malt used in the production.

To obtain a beer with a full bodied beer taste, one needs to have reasonable malt content (Leskosek and Mitrovic 1994). This reasonable malt content will lead to high fermentable sugar which eventually leads to high alcohol content.

A method of removing some of the alcohol contents will allow the brewers use reasonable malt content from the point of view of flavor majority without suffering the risk of excessive alcohol. To obtain a good tasting low alcoholic beer, a good method of dealcoholization that will retain the flavor and fragrance components of the wine must be used.

Early known and simplest way to achieve reduction in alcohol formation during beer production relied on inhibited alcohol formation during brewing processes (Leskosek and Mitrovic 1994). This has been the most economical way but leads to undesirable changes in taste and flavor profile of the product.
Osmotic membrane distillation method is a novel process that uses membrane to remove the alcohol content of fermented beverages still retaining the flavor and fragrance components of the wine (Hogan et al., 1998). It is particularly suited for concentration of heat-sensitive products such as fruit and vegetable juice, deaethanolisation of wine, pharmaceutical products and biological (Narayan et al., 2002).

OMD uses a hydrophobic microporous membrane, which separates the two aqueous solutions, one being the feed or dilute solution and the other being the osmotic agent (OA) or brine solution.

Advantages of OMD

OMD offers major advantages in comparison with other processes (Hogan et al., 1998).

- This process is highly selective for the removal of alcohol relative to water because the vapor pressure of water over most alcoholic ferment is very nearly that over pure water.

- The lower temperature employed can help avoid chemical reactions associated with heat treatment and prevent degradation of flavor, color and loss of volatile aroma.

- Only volatile compound which can permeate the membrane will be separated and non-volatile solutes such as ions, sugars, macro molecules, cells and colloids are totally retained in the feed.

- The transport rate of flavor/fragrance components from wine to strip solution is reduced because the solubility of these components in alcohol/water solution are substantially higher (and their vapor pressure lower) than they are in plain water.

The aim of this work was to study the effect of process parameters such as temperature, stirring speed and membrane pore size on the dealcoholization of beer (Star) and to fit a model equation that will explain the process.

MATERIALS AND METHODS

Beer

A star lager beer manufactured by Nigerian Breweries Plc. was bought from a local market at Abakpa Nike, Enugu, Enugu State Nigeria.

Membrane

A circular hydrophobic polytetrafluoroethylene (PTFE) membrane of 0.45μm and 0.25μm porosities and 142mm in diameter were bought from Sartorius Stedium Biotech Germany.

Distilled Water

Extra pure distilled water was bought from pyrogen free Water Company Setdeo Nigeria Limited New Haven Enugu, Enugu State, Nigeria.

Determination of Alcohol Content

The alcohol determination was done using distillation method, followed by specific gravity determination according to Brewing Process: EBC method.

Flux Determination

This was done using the method described by A.V. Narayan et al. The flux determination apparatus consisted of two glass reservoirs of equal volume (2L) labeled A and B. A was the feed reservoir with side arm at the base and was connected to osmotic membrane cell B with side arm at the base through a teflon tube. A known quantity of beer was introduced into A through the open vent and beer flowed by hydrostatic pressure into B until the sample in reservoir B touched the membrane unit.

A magnetic bar was introduced into reservoir B and the reservoir was mounted on the magnetic stirrer hot place. Reservoir A was equally mounted on a hot plate so that the two reservoirs were on the same level to cancel the effect of hydrostatic pressure changes due to difference in levels. The two reservoirs were placed on the same temperature.
In reservoir B, distilled water was introduced through the upper vent so that there were two fluid compartments (water and wine) separated by the membrane unit and the vent in B closed tightly to arrest vaporization.

The rise in height of water in cell side was measured every 1 hr for 4 hours with the aid of a meter rule attached to the upper part of reservoir B. The corresponding flux was calculated.

The system was subjected to different variables according to the design layout in standard order generated by the Design – expert software in Table 2 and their corresponding flux were calculated. Table 2 shows the factors and levels of two factorial design employed in the experiment.

**Table 1**: Factors and Levels of Full Factorial Design.

<table>
<thead>
<tr>
<th>FACTORS</th>
<th>UNITS</th>
<th>LOW LEVEL (-)</th>
<th>HIGH LEVEL (+)</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Temperature</td>
<td>°C</td>
<td>30</td>
<td>40</td>
</tr>
<tr>
<td>• Stirring Speed</td>
<td>Rpm</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>• Membrane Pore Size</td>
<td>µm</td>
<td>0.2</td>
<td>0.45</td>
</tr>
</tbody>
</table>

**RESULTS**

**Table 2**: Factorial Design and Corresponding Flux Values.

<table>
<thead>
<tr>
<th>Standard Order</th>
<th>Run Order</th>
<th>Factor 1 Temp (°C)</th>
<th>Factor 2 Stir Speed (rpm)</th>
<th>Factor 3 membrane Pore size (µm)</th>
<th>Response flux (l/m²·hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1</td>
<td>40</td>
<td>100</td>
<td>0.2</td>
<td>1.809</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>30</td>
<td>100</td>
<td>0.2</td>
<td>1.658</td>
</tr>
<tr>
<td>8</td>
<td>3</td>
<td>40</td>
<td>100</td>
<td>0.45</td>
<td>1.653</td>
</tr>
<tr>
<td>7</td>
<td>4</td>
<td>30</td>
<td>100</td>
<td>0.45</td>
<td>1.470</td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>40</td>
<td>0.00</td>
<td>0.2</td>
<td>1.421</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>40</td>
<td>0.00</td>
<td>0.45</td>
<td>1.386</td>
</tr>
<tr>
<td>5</td>
<td>7</td>
<td>30</td>
<td>0.00</td>
<td>0.45</td>
<td>1.194</td>
</tr>
<tr>
<td>1</td>
<td>8</td>
<td>30</td>
<td>0.00</td>
<td>0.2</td>
<td>1.356</td>
</tr>
</tbody>
</table>
DISCUSSION

The results obtained from the eight experiments performed according to the full factorial design (Table 2) were analyzed using Design-Expert software version 8.0.2®.

The half normality plot in Figure 1 which was used to select the effects that were included in the model showed that the main effects temperature, stirring speed and membrane pore size were significant since they lied on the right hand side of the line, their contribution has a positive effect on the model. The rest of the effects lied along the line were negligible.

The factor stirring speed B had the largest effect because it lied furthest from the line, followed by temperature A and the least being membrane pore size C.

The Pareto Chart (Figure 2) which was used to visualize the magnitude of the each effect equally had the same conclusion with half normality plot.

The ANOVA for the selected factorial model (Table 3) confirmed that the model is significant.

The model F – value of 46.62 implied that the model is significant. Values of "Prob > F" less than 0.0500 indicated that the model terms are significant. In this case A, B, C are significant model terms. Values greater than 0.1000 indicate the model terms are not significant.

The final model equation in terms of coded factors:

\[ \text{Flux} = +1.49 + 0.074\times A + 0.15\times B - 0.068\times C \] (1)

Final model Equation in terms of actual factors.

\[ \text{Flux} = +0.99795 + 0.014775\times \text{Temperature} +3.08250E-003\times \text{Stirring Speed} - 0.54100\times \text{pore size} \] (2)

Figure 1: Half Normal Plot – All Big Effects Selected.
Validation of Model

A good estimated regression model should explain the variation of the dependent variable in the sample. The model adequacy check done employed model diagnostic plots. Most of the plots displayed residuals which showed how well the model satisfied the assumptions of the analysis of variance.

Conclusion of Some of the Diagnostic Plots Used are:

- The normal probability plots followed a straight line.
- Plots of residuals vs. the ascending predicted response values showed a random scatter.

Table 3: ANOVA for Selected Factorial Model.

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F value</th>
<th>P - Value Prob &gt; F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>0.027</td>
<td>3</td>
<td>0.090</td>
<td>46.62</td>
<td>0.0014 significance</td>
</tr>
<tr>
<td>A – Temperature</td>
<td>0.044</td>
<td>1</td>
<td>0.044</td>
<td>22.59</td>
<td>0.0090</td>
</tr>
<tr>
<td>B – Stirring Speed</td>
<td>0.19</td>
<td>1</td>
<td>0.19</td>
<td>98.33</td>
<td>0.0006</td>
</tr>
<tr>
<td>C – Pore Size</td>
<td>0.037</td>
<td>1</td>
<td>0.037</td>
<td>1893.0</td>
<td>0.0121</td>
</tr>
<tr>
<td>Residual</td>
<td>7.730E – 003</td>
<td>4</td>
<td>1.933E – 0.003</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cor total</td>
<td>0.28</td>
<td>7</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 2: Pareto Chart.
• The plots of residuals vs. experimental run order showed a random scatter.

• The graph of predicted response values vs. actual response values split evenly by the 45 degree line.

• The residual analysis did not reveal any problems meaning that the model is valid and the assumptions made by ANOVA adequate.

**Temperature Effect**

The temperature effect showed that temperature at its higher level had higher mean response compared to that at low level. There was 8.34% increase on flux. This was because the slighted rise in temperature of the feed provided an additional driving force which in turn increased the activity coefficient of the feed solution (Narayan et al., 2002).

It is not difficult to appreciate the strong dependence of flux on temperature. It is known that mass transfer coefficient in many transport process shows Arrhenius dependency on temperature (Coulson and Richardson 2004). Similar behavior was observed on the present study due to the fact that the activity coefficient of the strip solution remained constant over the range of temperature studied here.

**Figure 3:** Single Effect of Temperature on Flux.
Stirring Speed Effect

This main analysis showed that stirring speed had greatest effect on flux with 21.4% increase.

Stirring provided mild agitation of the feed solution thereby reducing the effect of membrane fouling. Membrane fouling results from irreversible blocking of membranes by adhesion of insoluble compounds to the membrane matrix reducing the rate of flux. Essentially, stirring disturbs the concentration polarization layer near the membrane surface, enabling uniform concentration throughout the cell and thereby maintaining an effective driving force by the osmotic solution across the membrane.

Membrane Pore Size

The membrane pore size was varied (0.2 – 0.45) at various combination with other factors using the design layout. The single effect plot (Figure 5) showed that membrane pore size had a negative effect on flux with 86% decrease on flux. This was in line with the literature where it was stated that small diameter hollow fiber membrane with thin walls appears to be the best candidate for OMD because they offer high area to volume ratio and has high pore entry pressure (Polopvik and Riverol 2005). It was also stated that the characteristics of the membrane use for OMD is that it should be highly porous (60 – 80%) and as thin as possible (0.1 – 1µm) to meet the penetration – pressure limitation posed by the classical Kelvin equation where pore size is inversely proportional to the pore-entry pressure (David wollan 2007).

Interaction Effects of the Three Factors

Cubic plot was used to analyze the interaction effects of the three factors modeled. The cubic plot in fig 6 was used to get the combination of the levels of the factors that can give maximal flux.

![Figure 4: Single Effect of Stirring Speed on Flux.](image-url)
**Figure 5**: Single Effect of Membrane Pore Size on Flux.

**Figure 6**: Cube Plot of the Three Factors.
The flux was maximum at highest temperature of 40°C, highest stirring speed of 100 rpm and lowest pore size of 0.2 µm with a value of 1.789 at the upper front right corner of the cube. Moreover, the least flux was obtained at the lowest temperature of 30°C, lowest stirring speed of 0 rpm and highest pore size of 0.45 µm with a value of 1.198 at the lower back left corner of the cube.

CONCLUSION

The result of this present study confirmed the viability of applying OMD for dealcoholization of beer. Of all the factors investigated, stirring speed had highest effect on flux, followed by temperature; the least was membrane pore size.

- It was therefore concluded that flux is directly proportional to temperature and stirring speed but inversely proportional to the membrane pore size.

- The optimal settings that can give highest flux when applied are temperature of 40°C, stirring speed of 100 rpm and membrane pore size of 0.2 µm.

- It was also established that rise in water level which is flux is directly proportional to amount of alcohol removed.

- It was also established based on the statistical analysis done that the model equation obtained was appropriate in describing the dealcoholization process.

- All the results were obtained at the condition of the experiment.

RECOMMENDATIONS

- The problem of OMD is constraints of low flux. Attempt should be made to enhance transmembrane flux by application of acoustic field that will induce mild circulatory currents which disturbs the hydrodynamic boundary layer of the solution.

- The main draw backs of the industrial application of OMD are the feasibility of membrane reuse, and can be overcome by selecting the appropriate membrane (very resistant and long life) and carefully controlling the process parameters – temperature and stirring speed.

REFERENCES


SUGGESTED CITATION