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# Dealcoholisation of standard solutions by reverse osmosis and diafiltration

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## ABSTRACT

The methods of beer dealcoholisation can be divided in two large groups: physical and biological methods. One of the physical methods – reverse osmosis was chosen and studied. Four standard solutions were suggested for further research. These standard solutions contained basic substances of beer: water, ethanol and glucose. The effect of the substances added to the solution, and influence of retentate flow on the permeate flux were studied. The results of initial experiments were taken as the basis for further diafiltration process, where significant reduction of the ethanol concentration in the retentate was expected. Experimental results were compared with values calculated from mathematical model found in the cited literature. It was found out that the experimental data agree very well with calculated model data. During long-time process of diafiltration the concentration of ethanol decreased from the initial value 4.49 to 0.24 vol%. The studied diafiltration process is suitable for the dealcoholisation of investigated mixtures.

*Keywords:* Dealcoholisation; Standard solutions; Membrane processes; Reverse osmosis; Beer

## 1. Introduction

The basic motive for drinking non-alcoholic beverages is well known. Consumption of alcoholic beverages before driving is not accepted in many countries around the world. Content of ethanol in alcoholic beverages is determined by value of maximum alcohol by volume (ABV). This value is established by legal regulation in different countries. Beer with low alcohol content can be divided into two subcategories in the EU countries. Beers called low-alcohol beers are included in the first subcategory. ABV content is less or equal then 1.2. The second subcategory is classified as an alcohol-free beer (≤ 0.5 ABV) [1]. The other biggest reason is increasing interest of consumers in health and weight management [2].

The main point of the beer dealcoholisation is partial removal of ethanol. First attempts were not very successful. Ethanol was removed but the obtained beverage was different compared with alcoholic beer. The taste, flavour and colour were changed. After that new methods for beer dealcoholisation were evolved. These new approaches are focused on the attainment of flavour balance between volatile and non-volatile compounds, and ethanol [3].

Methods for alcohol-free beer production can be generally divided into two large groups; biological and physical methods [1]. Biological methods are used during the process of beer production. They are focused on the prevention or restriction of alcohol formation [4,5]. Advantages of biological methods are obvious. They can use existing technology and operating costs are maintained [3].

As mentioned earlier, whole fermentation process is important for great aroma profile of beer. Biological methods are known as methods which interrupt the fermentation process. The resulting aroma profile of beer is damaged [6–9].

Physical methods offer possibility for a good aroma profile. They are based on the removal of ethanol from the final

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product. Thermal and membrane processes are included in this category. Ethanol can be completely removed with thermal processes [3]. But they also have some disadvantages. One of them is negative influence on aroma profile of the product. The other disadvantages are cost of this technology and high energy consumption [4].

A second group of physical methods consist of membrane processes which can be a good substitution in this field. Membrane processes operate at optimal conditions, thus preserving the sensory characteristics of the original product and their energy consumption is lower compared with classical operations [10]. Pervapouration [11], dialysis and reverse osmosis (RO) are the most investigated membrane processes.

Pervapouration is classified as one of the best processes for beer aroma recovery. Combination of pervapouration and special physical methods provides good results and could be a suitable process for alcohol-free beer production [12–15]. The other known membrane process is dialysis. The utilisation of dialysis was popular at 1980s–1990s [16,17].

The last used process is RO [11,18]. It is the process used in desalination of seawater (or brackish water) basically. Water as low molecular solvent is separated from solution with various type of solutes (e.g., univalent, monovalent ions) [19].

RO in batch diafiltration mode was investigated in our research. Diafiltration process is unique procedure for purifying a multisolute system [20]. Purification is performed by adding diafiltration liquid (in our case demineralised water). Solutes are divided into two main groups: high molecular weight and low molecular substances [21]. Specification of these substances is highly dependent on the specific type of separation process. Proteins, saccharides, molecules of dyes are perceived as the high molecular substances, but multivalent and monovalent ions are rejected by RO also [19]. Low molecular substances are molecules of water (or solvent, ethanol was used in our measurements). The following mathematical model (Eq. (1)) describes our type of diafiltration mode [19,22,23]. This model is based on a few assumptions: rejection factor of high molecular substances is equal to 100%, volume of the solution in the storage stirred tank remains steady and rejection factor of low molecular substance is constant. Mathematical model was modified for our purposes.

$$c_{EtOH,F} = c_{EtOH,F,0}\, e^{-\frac{J_P A\tau\left(1-R_{etOH}\right)}{V_T}} = c_{EtOH,F,0}\, e^{-\frac{V_{DL}\left(1-R_{etOH}\right)}{V_T}} \qquad (1)$$

where $c_{\mathrm{EtOH},F}$ is current concentration of ethanol in feed, $c_{\mathrm{EtOH},F,0}$ is starting concentration of ethanol in feed, $V_T$ presents volume of solution in stirred tank and $V_{\mathrm{DL}}$ is volume of used diafiltration liquid.

Our research was divided into three main aims. Firstly, the effect of the substances added to the standard solution on the permeate flux at different operating conditions was investigated. The second aim of the interest was the influence of the operating conditions on the rejection of substances. The last goal was a comparison of experimental data and selection of the best conditions for the RO in the diafiltration mode.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Membrane

The membrane used was spiral-wound industrial brackish water RO element commercialised by GE Power & Water, Pennsylvania, USA (Model: SG2540F30) with the following characteristics provide in Table 1 [24].

#### 2.1.2. Standard solutions

Four standard solutions were proposed in this work. The volume of each model solutions ($V_T$) was 40 L. Substances, which were added in these solutions, are the basic components included in beer (ethanol and glucose). Demineralised water and drinking water which contain minerals were used during the process (both types of used water were characterised by conductivity κ).

### 2.2. Analytical methods

The selected analytical methods were applied, mainly because of their feasibility. The analytical methods which were used are presented in Table 2.

Table 2
Information about applied analytical methods

| Analysed substances | Origin of the sample | Analytical method |
|---|---|---|
| Ethanol | Feed, permeate, retentate | Gas chromatography |
| Glucose | Feed, permeate, retentate | Polarimetry |
| Contents of salts | Feed, permeate, retentate | Conductivity meter |

Table 1
Characteristics of the membrane used during measurements

| | | | | |
|---|---|---|---|---|
| Model | SG2540F30 | Maximum operating pressure | $T < 35$°C | 600 psi |
| Material | Polyamide | | $T > 35$°C | 435 psi |
| Active area | 2.6 m$^2$ | pH range | 5.5–7 (optimum rejection) | |
| Average permeate flow | 2.2 m$^3$ d$^{-1}$ | | | |
| Average NaCl rejection | 98.5% | | 2.0–10.0 (continuous operation) | |
| Minimum NaCl rejection | 97% | | | |
| Maximum temperature | 50°C (continuous operation) | | | |

### 2.2.1. Gas chromatography conditions

Content of ethanol was measured on Gas Chromatograph SHIMADZU GC-2014. Fused silica capillary Supelcowax-10 (15 m × 0.32 mm × 0.5 µm) was used.

### 2.3. Experimental setup and methods of experiments

The apparatus depicted in Fig. 1 was used for the experiments.

During measurements some process parameters were varied:

- Pressure difference: 10, 15 and 20 bar.
- Retentate flow: 600 and 800 L $h^{-1}$.
- Temperature was maintained constant at the value of 20°C.

Four standard solutions were individually prepared according to Table 3. Each standard solution (5) was pumped from the stirred storage tank (1) to the membrane module (4). Operating pressure was adjusted by reducing valve. After setting parameters and stabilisation of the process, time needed to obtain 0.5 or 1 kg of permeate (6) (depending on the model solution) was measured. Measurements were repeated three times for each setting. Samples were taken from feed, permeate and retentate. Diafiltration process was carried out under selected conditions. Experiment started after the process of stabilisation and sampling. Approximately, 3 L of permeate were continuously removed. This amount was replenished by adding demineralised water back to the stirred storage tank. Sampling was carried out in half-an-hour intervals and the content of salts was determined using conductivity measurements. Ethanol and glucose were later analysed in laboratory.

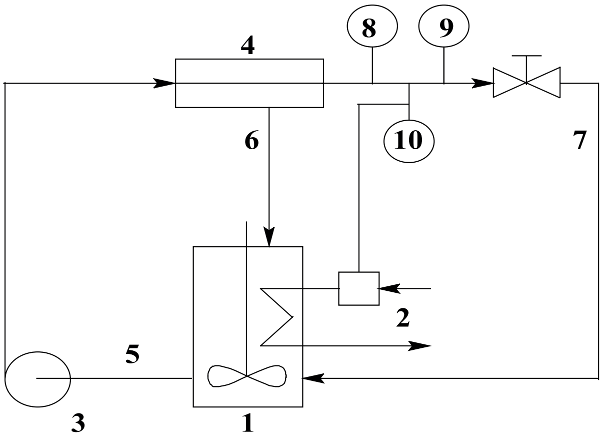

Fig. 1. Diagram of the apparatus: 1 – stirred tank; 2 – water cooling system; 3 – diaphragm pump; 4 – membrane module; 5 – feed; 6 – permeate; 7 – retentate; 8 – flow meter; 9 – pressure transducer and 10 – thermometer.

Table 3
Substances included in standard solutions

| $V_T = 40$ L | $\kappa_{\text{DEMI. WATER}} =$ 6.6 µS $cm^{-1}$ | $\kappa_{\text{DRINK. WATER}} =$ 564 µS $cm^{-1}$ |
|---|---|---|
| Standard solution A | Demineralised water | Glucose, $m = 1.5$ kg |
| Standard solution B | Drinking water | Glucose, $m = 1.5$ kg |
| Standard solution C | Demineralised water | Ethanol, $V = 2$ L |
| Standard solution D | Drinking water | Glucose, $m = 1.5$ kg |

Note: Demineralized water and drinking water is specified by conductivity of water.

## 3. Results and discussion

Firstly, characterisation of membrane was performed. The main goal of these experiments was verification of values of sodium chloride rejection declared by the membrane producer. Measured values were in a good agreement with values declared by membrane producer.

### 3.1. Influence of substances added to the solution and retentate flow on the permeate flux

Firstly, two standard solutions were prepared, standard solution A (demineralised water, glucose) and standard solution B (drinking water, glucose). Influence of glucose addition to the solution on the permeate flux is shown in Fig. 2. The permeate flux decreased considerably in comparison with permeate flux of demineralised water. This indicates influence of osmotic pressure of glucose. There is a minimum difference between permeate flux of standard solution B and solution A. It follows that the used type of water has only a slight influence on the permeate flux. The second investigated parameter was effect of the retentate flow. Each of standard solutions was studied under two settings of retentate flow, 600 and 800 L $h^{-1}$. It was observed that higher retentate flow provides better results of permeate flux and rejection factor (this trend was observed in all measurements). That can be caused by decrease of concentration polarisation. This phenomenon is connected with relations of feed solution and membrane, close to the membrane. Solutes are accumulated on the membrane surface and it leads to decrease of permeate flux. The limit concentration of the solutes on the membrane surface can create filter cake or gel layer [19]. It cause increase osmotic pressure within the formed layer of solutes on the

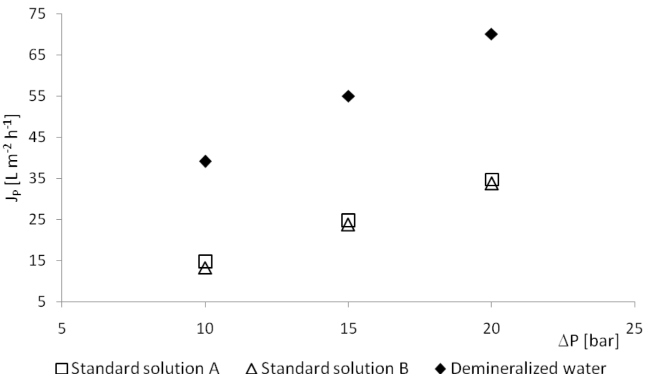

Fig. 2. Dependence of the permeate flux on the pressure difference; retentate flow of 600 L $h^{-1}$.

surface of membrane also [25]. There are a lot of methods which can be used for lowering of concentration polarisation during membrane processes [26–28].

One of these methods is focused on the effect of process conditions. Reducing of the concentration polarisation is achieved by increasing the mass transfer coefficient. This increasing can be provided by growing feed flow velocity [29]. Van Gauwbergen and Baeyens [30] investigated macroscopic fluid flow conditions in spiral-wound membrane elements. Moreover, they confirmed the positive effect of the higher feed flow on the reduction of the concentration polarisation.

Comparison of all standard solutions is shown in Fig. 3. Standard solutions A and B are known from the former text. The next model solutions were standard solution C (demineralised water, ethanol) and standard solution D (drinking water, glucose and ethanol). The interesting results are obtained by comparing permeate fluxes for standard solution A, standard solution C and demineralised water. Experiments show that the significant reduction of the permeate flux is caused by ethanol. Probably, this decrease is caused by osmotic pressure of ethanol. Because of the precious values of osmotic pressure require knowledge of unknown transport model parameters [31] for selected membrane process, these values were not determined.

Following decrease of permeate flux follows from mixing all components in standard solution D. Although the difference between standard solution B and D is only in addition of ethanol and drinking water, experimental results show considerable decrease of permeate flux.

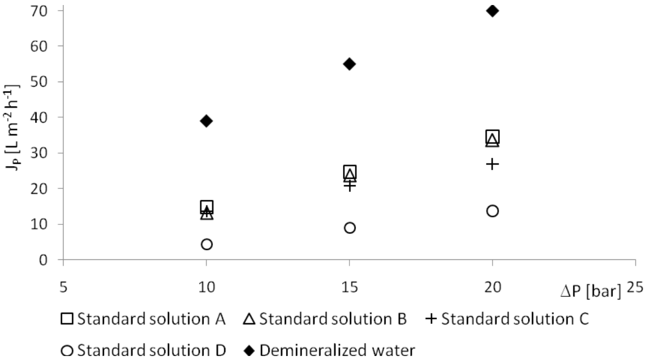

Fig. 3. Dependence of the permeate flux on the pressure difference; retentate flow of 600 L h$^{-1}$.

### 3.2. Operation of RO in diafiltration mode experiments

Previous experiments were the basis for the following step, which was the diafiltration. Significant reduction of ethanol concentration in the retentate was expected. The best conditions for this process were selected from realised experimental results. Analysis of these results showed, that the best conditions for diafiltration process are: standard solution D, pressure difference ($\Delta P$) 20 bar, retentate flow ($\dot{V}_R$) 800 L h$^{-1}$.

Tables 4 and 5 represent the results from experiments. Sampling was carried out in half-an-hour intervals ($\tau$). The volume of used diafiltration liquid is shown in the second columns. The concentrations of substances obtained during the experiments are also included ($c_{GL}$; $c_{EtOH}$). Rejection parameters are calculated for glucose ($R_{GL}$) and ethanol ($R_{EtOH}$). Table 4 shows experimental results for retentate and concentrations of ethanol calculated from the mathematical model ($c_{EtOH,M}$) are presented there. Permeate flux ($J_P$) can be found in Table 5.

One of the interesting findings was observed during the process. Permeate flux increases during the experiment (Table 5). It indicates the removal of ethanol from standard solution D ($c_{EtOH,R}$ in Table 4), while the concentrations of other substances were maintained. The second interesting finding is focused on permeate flux also. The values of permeate flux of standard solution B and standard solution D in the end of the RO in diafiltration mode under the same

Table 5
Results of diafiltration experiments for permeate

| $\dot{V}_R$ = 800 L h$^{-1}$ | | $\Delta P$ = 20 bar | | $T$ = 20°C ± 0.3°C |
|---|---|---|---|---|
| $\tau$ (h) | $V_{DL}$ (L) | $c_{GL,P}$ (g L$^{-1}$) | $c_{EtOH,P}$ (vol%) | $J_P$ (L m$^{-2}$ h$^{-1}$) |
| 0 | 0 | 0.17 | 3.07 | 14.14 |
| 0.5 | 21 | 0.17 | 1.98 | 17.15 |
| 1 | 48 | 0.17 | 1.27 | 21.42 |
| 1.5 | 75 | 0.18 | 0.70 | 24.42 |
| 2 | 111 | 0.16 | 0.40 | 26.78 |
| 2.5 | 147 | 0.17 | 0.22 | 28.72 |
| 3 | 183 | 0.16 | 0.16 | 29.36; 34.47[a] |

[a] Permeate flux for standard solution B under conditions: $\dot{V}_R$ = 800 L h$^{-1}$; $\Delta P$ = 20 bar; $T$ = 20°C ± 0.3°C.

Table 4
Results of diafiltration experiments for retentate

| $\dot{V}_R$ = 800 L h$^{-1}$ | | $\Delta P$ = 20 bar | $T$ = 20°C ± 0.3°C | | $\bar{R}_{EtOH,M}$ = 32.70% | |
|---|---|---|---|---|---|---|
| $\tau$ (h) | $V_{DL}$ (L) | $c_{GL,R}$ (g L$^{-1}$) | $c_{EtOH,R}$ (vol%) | $c_{EtOH,M}$ (vol%) | $R_{GL}$ (%) | $R_{EtOH}$ (%) |
| 0 | 0 | 35.16 | 4.49 | 4.49 | 99.52 | 31.63 |
| 0.5 | 21 | 37.95 | 2.97 | 3.15 | 99.52 | 33.33 |
| 1 | 48 | 38.88 | 1.82 | 2.00 | 99.52 | 30.22 |
| 1.5 | 75 | 39.81 | 1.07 | 1.27 | 99.51 | 34.58 |
| 2 | 111 | 39.81 | 0.60 | 0.69 | 99.54 | 33.33 |
| 2.5 | 147 | 40.28 | 0.37 | 0.38 | 99.52 | 40.54 |
| 3 | 183 | 39.81 | 0.24 | 0.21 | 99.54 | 33.33 |

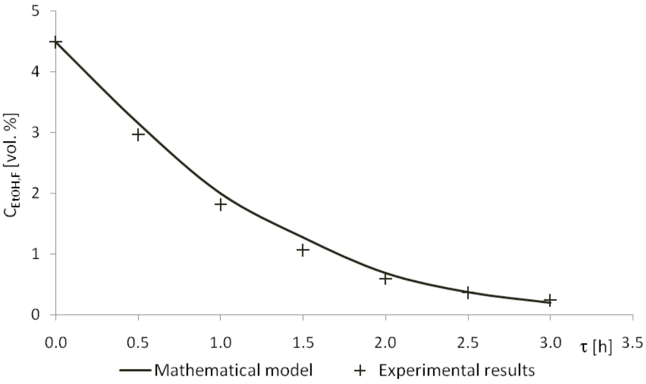

Fig. 4. Comparison of experimental results and calculated data.

conditions ($\dot{V}_R$; $\Delta P$ and $T$) were compared. It was proven that the final value of permeate flux for standard solution D is 17% less than the value of permeate flux for standard solution B. It also illustrates a removing of ethanol during the process.

It was mentioned earlier, the mathematical model of batch diafiltration was found in the literature. The values of ethanol concentration in the retentate flow ($c_{\mathrm{EtOH},M}$) were calculated in compliance with the conditions listed in section with theoretical background (Eq. (1)). The rejection factor for ethanol gently fluctuated during the diafiltration process. The average value of rejection factor ($\bar{R}_{\mathrm{EtOH},M}$) for ethanol was used in the calculations. The ethanol concentrations in retentate flow evaluated from the mathematical model ($c_{\mathrm{EtOH},M}$) were compared with experimental results ($c_{\mathrm{EtOH},R}$). Comparison of experimental results and calculated values is shown in Fig. 4. It was observed that the experimental data ($c_{\mathrm{EtOH},R}$) agree very well with calculated model data ($c_{\mathrm{EtOH},M}$) (Table 4).

## 4. Conclusions

The present paper deals with a brief review of methods for alcohol-free beer production and with use of one of them for dealcoholisation process. The main issue of beer dealcoholisation is a loss of aroma and flavour. This problem is possible to be solved using membrane technology. Membrane processes operate at optimal conditions, thus preserving the sensory characteristics of the original product. RO and RO in diafiltration mode were selected as a studied membrane processes in our research.

Four standard solutions were proposed in this work. Substances, which were added into these solutions, are the basic components included in beer: ethanol, glucose and water. These standard solutions were separated by RO. Firstly was investigated the effect of the substances added to the solution and influence of the retentate flow on the permeate flux and rejection parameter. Enhancement of the permeate flux with increasing of the pressure difference was observed. However, permeate flux of the standard solutions considerably decreased in comparison with permeate flux of demineralised water. Evaluating of investigated substances effect on the decreasing permeate flux show that the biggest negative influence on this process has ethanol. The other studied factor was the influence of the retentate flow on the permeate flux and the rejection parameter. The higher values of the retentate flow provided slightly better results of permeate flux and rejection parameter.

The second aim of the interest was the influence of the operating conditions on the rejection parameter of the substances. The higher retentate flow and upper applied pressure difference provided the higher rejection parameters for all substances.

These results of experiments were the basis for the next step, which was the RO in batch diafiltration mode. The best conditions for this process were selected from previous experimental results. Analysis of the previous results showed that the best conditions for diafiltration mode are: model solution D, retentate flow 800 L h$^{-1}$ and pressure difference 20 bar. In this process was expected significant reduction of the ethanol concentration in the retentate. Experimental results were compared with values calculated from mathematical model. It was found out that the experimental data agree very well with calculated model data. During long-time process of diafiltration the concentration of ethanol decreased from the initial value 4.49 to 0.24 vol%. The studied diafiltration process is regarded as suitable for the dealcoholisation of investigated systems.

## Acknowledgement

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## Symbols

| | | |
|---|---|---|
| ABV | — | Alcohol by volume |
| $c_{\mathrm{EtOH},F}$ | — | Current concentration of ethanol in feed |
| $c_{\mathrm{EtOH},F,0}$ | — | Starting concentration of ethanol in feed |
| $c_{\mathrm{EtOH},M}$ | — | Concentration of ethanol calculated from mathematical model |
| $c_{\mathrm{EtOH},P}$ | — | Concentration of ethanol in the permeate |
| $c_{\mathrm{EtOH},R}$ | — | Concentration of ethanol in the retentate |
| $c_{\mathrm{GL},P}$ | — | Concentration of glucose in the permeate |
| $c_{\mathrm{GL},R}$ | — | Concentration of glucose in the retentate |
| DEMI | — | Demineralized water |
| DRINK | — | Drinking water |
| $J_P$ | — | Permeate flux |
| $\kappa$ | — | Conductivity of water |
| $\Delta P$ | — | Pressure difference |
| $R_{\mathrm{GL}}$ | — | Rejection parameter for glucose |
| $R_{\mathrm{EtOH}}$ | — | Rejection parameter for ethanol |
| $\bar{R}_{\mathrm{EtOH},M}$ | — | Average value of rejection factor of ethanol; used in the calculations |
| $\tau$ | — | Time of the experiment and sampling |
| $V_{\mathrm{DL}}$ | — | Volume of used diafiltration liquid added during the process |
| $V_T$ | — | Volume of model solution in stirred tank |
| $\dot{V}_R$ | — | Retentate flow |

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